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The Influence of Drilling-Fluid Rheology on Cuttings-Bed Behavior

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Doctoral thesis

NTNU
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Thesis for the Degree of
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Summary

Transporting cuttings out of the wellbore is an important part of every drilling operation to ensure efficiency and the reduction of non-productive time. The drilling fluids used for this task are complex fluid systems, generally with either oil or water as a base substance. The hole-cleaning performance of these two fluid systems is reportedly different from each other. Industry experience has shown that oil-based drilling fluids are performing better than water-based drilling fluids, even when the viscosity is similar. Additionally, research results reported in the literature show diverse conclusions with superior behavior for either water-based or oil-based fluids, or findings where neither of the types excelled. A general conclusion has not been made and the reasons for the different behavior are not entirely understood.

The present thesis investigates the influence of the viscoelastic properties of drilling fluids on cuttings transport and hole cleaning. For this purpose an extensive rheometer study was conducted to measure the rheological properties of drilling fluids. Flow-loop experiments compare the performance of these fluids regarding their hole cleaning and cuttings-transport abilities in a controlled experimental setup. The tested fluids were three oil based and one water-based drilling fluid. The oil-based fluids were water-in-oil emulsions with a yield stress (named OBM A, B, and C), containing barite, $CaCl_2$, bentonite, lime, emulsifier, and a fluid loss agent. The water-based fluid was a KCl fluid, viscosified with xanthan gum. Other additives were glycol, polyanionic cellulose, starch, soda ash and barite. OBM B and the KCl fluid were adjusted to have a similar viscosity profile in the relevant shear-rate range of the flow-loop experiments. The fluid composition and description is presented in chapter 2.2.

The rheological investigation involved a preconditioning study, measurement of high shear rate and low shear-rate flow curves, measurement of temperature dependence, amplitude-sweep tests, shear-stress sweep tests and the measurement of thixotropy. A detailed description of the measurement parameters is given in chapter 2.1. The experiments were carried out at the shared fluid laboratories of SINTEF Petroleum and the Department of Geoscience and Petroleum at NTNU in Trondheim, and partly at SINTEF Petroleum in Bergen. The flow-loop experimental rig had a 10 m long test

section with a fully eccentric rotating drill string. Experiments were performed at varying fluid velocities and with or without drill-string rotation.

Chapter 2.3 presents the main results of the rheological analysis and the flow-loop experiments. In a direct comparison between OBM B and the KCl fluid, the OBM B performed better and removed more sand out of the test section than the KCl fluid, for experiments without drill-string rotation. Drill-string rotation demonstrated dominating behavior over the flow related properties regardless of the rotational velocity. The viscoelastic properties were found to only have a small impact on the cuttings transport, but rather to influence the cutting beds resistance to erosion. An about 100 times higher strain tolerance was found for the KCl fluid in amplitude-sweep tests, compared to OBM B. This higher elasticity increased the cuttings-bed resistance to erosion and created a stronger connection between the cutting particles. Comparably, the yield stress in the OBM B was broken more easily than the elasticity in the KCl fluid, leading to more efficient hole cleaning for OBM B compared to the KCl fluid.

Other rheological results indicated a strong time dependence of the fluid properties. The thixotropy measurements displayed a structural recovery, which is exceeding the structure of the initial rest interval by up to 160 %. The time dependent structural changes of the OBMs were also investigated in a preconditioning study to quantify the effect of pre-shearing and waiting time on flow curves and amplitude sweeps. The effects of pretreatment are influencing the reproducibility of the results. Establishing a strict measuring routine is therefore recommended as the results are expected to be applicable to most oil-based drilling fluids.

The main results of the present PhD-thesis are presented and discussed as follows:

- Rheological results in chapter 2.3
- Flow-loop results in chapter 2.4

Detailed experimental studies and results are presented in five articles in the appendix.

Foreword

The research work is a project initiated by Sintef Petroleum and supported by the Research Council of Norway, Statoil and Aker BP. The work was mainly conducted at NTNU and Sintef Petroleum in Trondheim and Bergen. Two PhD candidates were involved in the research work, where the current thesis presents the rheological investigation, and the complete flow-loop experiments can be found consulting (Sayindla et al. 2017). As the two parts of the project were intertwined, also the work responsibilities were tightly connected.

The preparation before the start of the flow-loop experiments was filled with time intensive tasks. A modification of the existing flow loop to accommodate for oil-based fluids was made, a new filter system was installed, load cells were set in place, concrete segments for the test section repaired and inserted.

The rheological experiments were conducted in parallel to the flow-loop experiments.

My contribution to the present project was:

- Modification and assembly of the flow loop prior to experiments
- Cleaning and maintenance of the flow loop between and after the experimental campaigns
- Preparation and monitoring of the drilling fluids
- Planning and conducting rheological experiments with Anton Paar rheometer and Fann 35 viscometer
- In-depth analysis of the rheology data
- Analysis of the connection between the rheological properties and the flow-loop results

List of Publications and Contributions

The following technical articles were presented at different conferences and published in conference proceedings and journals during the work for the thesis. My contribution in terms of experimental work, analysis and writing is stated in parenthesis:

- Paper 1, "Establishing an Experimental Preconditioning Procedure for Rheological Characterization of Oil-Based Drilling Fluids" focuses on the pretreatment of oil-based drilling fluids before the measurement of flow curves and amplitude sweeps with Fann 35 viscometers and Anton Paar rheometers. The paper was presented by Velaug Myrseth at the Nordic Rheology Conference in Karlstad and published in the annual transactions of the Nordic Rheological Society, Vol. 23, 2015 (10%/40%/40%)

- Paper 2, "Rheological Properties of Oil-Based Drilling Fluids and Base Oils" presents the rheological experiments and analysis of the OBMs A and B. The paper was presented at the International Conference on Ocean, Offshore and Arctic Engineering in St. Johns 2015 and published in the conference proceedings. (90%/50%/50%)

- Paper 3, "Effects of Oil-Based Drilling Fluids Rheological Properties on Hole-Cleaning Performance" presents the rheological experiments and analysis of OBM C and compares them to the results of OBM A and B. The rheological data is used to predict the pressure drop in the flow-loop experiments. The paper was presented at the International Conference on Ocean, Offshore and Arctic Engineering in Busan 2016 and published in the conference proceedings.
(90%/60%/70%)

- Paper 4, "Viscoelastic Properties of Drilling Fluids and their Influence on Cuttings Transport" compares the rheological properties of OBM B and the KCl fluid. The results of the rheological investigation are set into relation to the findings from the hole-cleaning experiments in the flow loop to find out if there is a connection to the viscoelastic properties. The article was accepted by the Journal of Petroleum Science and Engineering and published in volume 156 in July 2017. (95%/80%/80%)

- Paper 5, "Effect of Preconditioning and Ageing on Rheological Properties of Model Drilling Fluids" is a continuation of paper 1. Here water-based fluids with different amounts of salt and xanthan gum are analysed to conclude on the effects of pre-shearing and waiting time. The paper was presented as a poster by Bjørnar Lund at the Annual European Rheology Conference in Copenhagen and published in the annual transactions of the Nordic Rheological Society, volume 25, 2017 (10%/80%/80%)

Other relevant contributions in publications:

- Ytrehus, Jan David; Taghipour, Ali; Sayindla, Sneha; Lund, Bjørnar; Werner, Benjamin; Saasen, Arild. (2015) Full Scale Flow Loop Experiments of Hole Cleaning Performances of Drilling Fluids. Proceedings ASME 2015 34th International Conference on Ocean, Offshore and Arctic Engineering Volume 10: Petroleum Technology.

- Sayindla, Sneha; Lund, Bjørnar; Taghipour, Ali; Werner, Benjamin; Saasen, Arild; Gyland, Knud Richard; Ibragimova, Zalpato; Ytrehus, Jan David. (2016) Experimental investigation of cuttings transport with oil based drilling fluids. ASME 2016 35th International Conference on Ocean, Offshore and Arctic Engineering - Volume 8: Polar and Arctic Sciences and Technology; Petroleum Technology.

Acknowledgement

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1. Introduction

1.1 Background

Drilling operations to reach hydrocarbon reservoirs are challenging technical processes. The state of the art technology involves drilling rigs, a rotary drill bit connected to a drill string and a drilling-fluid circulation system. Compressional and torsional forces applied to the rotating drill bit lead to penetration of the rock. While drilling continues it becomes necessary to stabilize the borehole walls with casings to protect them from collapsing.

Penetrating the rock formation with the drill bit, creates rock fragments of varying sizes, called cuttings. These cuttings have to be removed out of the borehole to avoid the drill string to get stuck. For this reason a drilling fluid is pumped down through the inside of the drill string and exits at the drill bit. The cuttings are suspended in the drilling fluid in the annulus and are pumped up to the drilling rig. In the fluid-surface circulation loop the cuttings are removed by a separation process preparing the fluid for reuse. The first step of the cleaning process is the shale shaker which removes coarse rock cuttings while the slurry passes over vibrating screens. Finally, the clean drilling fluid is collected in a suction pit and ready to be pumped back into the well, or treated with chemicals to adjust properties.

Transporting cuttings out of the borehole is not the only task of drilling fluids. Providing borehole stability in uncased wellbore sections, cooling and lubricating the drill bit and the drill string, assuring formation integrity, and transmitting hydraulic energy to downhole tools are other important functions (Nazari, Hareland, and Azar 2010).

Drilling fluids have either water, brine, or refined oil as a base fluid (Caenn and Chillingar 1996). Commonly the water-based fluids can be divided into bentonite systems and KCl/Glycol systems. The base fluid is enriched with several components. Viscosifiers, such as clay or polymers increase the viscosity, barite is used as a weight material to increase the density, and lye can be added to control the pH-value. Other additives will depend on the rock formation, the wellbore conditions, and the drilling parameters. The composition of the fluids determines the fluids properties. Generally the emulsion in an oil-based drilling fluid results in a relatively high yield stress, a low gel strength at the micro level, and a low elasticity. In water-based fluids the polymers exhibit a lower yield stress, but a higher gel strength at the micro level and an

apparently high elasticity. However, the clay platelets in bentonite fluids, are resulting in a relatively high yield stress and gel strength, but exhibit a lower elasticity.

Oil-based and water-based drilling fluids each have advantages and disadvantages. Oil-based drilling fluids provide a better lubrication for the drill string, higher boiling points and lower freezing points, a high performance and capacity for reuse, better borehole stability, and are easier to maintain. In contrast these are more prone to lost circulation and are considered less environmentally friendly than water-based drilling fluids. The disadvantages of water-based drilling fluids are the ability to corrode metals, due to the added salts, and the lower inhibition against clay swelling. Water-based drilling fluids are more problematic to use at higher temperatures. Their benefits are easier control of viscosity and density, lower environmental impact (Apaleke, Al-Majed, and Hossain 2012), and lower costs.

All the mentioned properties and possibilities of fluid composition affect the performance of the drilling fluids during drilling operations. The hole cleaning and cuttings-transport capabilities of the drilling fluids are some of the most important issues when selecting a fluid of a particular well section. Additionally cost and environmental considerations have to be taken into account. Industry experience has shown that oil-based drilling fluids clean the borehole more effectively than water-based fluids. The reasons for this are not completely understood and the research literature is limited on controlled comparative studies where oil-based and water-based fluids with similar viscosities are tested. This is probably because the handling of oil-based drilling fluids in experimental facilities is challenging. This thesis investigates the influence of the viscoelastic properties of drilling fluids on cuttings transport and hole cleaning. The results will contribute to the understanding of drilling-fluid behavior and cuttings transport, to develop more efficient drilling-fluid systems.

1.2 Hole Cleaning and Cuttings Transport

Hole cleaning describes the process of removing drilled cuttings from the wellbore and transporting them to the surface. The parameters influencing the hole cleaning and cuttings transport can be divided into three groups, fluid parameters like density and viscosity, cutting parameters like cutting size and cutting concentration, and operational

parameters, like wellbore inclination and drill-pipe rotation (Bilgesu, Mishra, and Ameri 2007). An overview of cuttings-transport studies is given by Pilehvari, Azar, and Shirazi (1995) and more recently by Li and Luft (2014). Differences cannot only be seen by the afore mentioned parameters, different types of drilling fluids will affect the cuttings transport as well.

Industry experience shows oil-based and water-based drilling fluids are behaving differently in terms of hole cleaning and cuttings transport, even with similar viscosities and densities. Through recent years, different research groups have conducted experiments, or evaluated field data regarding cuttings transport and hole cleaning. Some groups also suggest reasons for the different behavior. The findings are diverse and reach from observations where oil-based drilling fluids perform better to results where water-based drilling fluids perform better to results where the cleaning difference between both types is low. The results reported by Hareland, Azar, and Rampesad (1993) show a better annular cleaning for water-based drilling fluids than oil-based drilling fluids for deviated holes from 40 ° to 50 °. Their experiments were conducted with an invert emulsion mineral oil-based drilling fluid and a water-based drilling fluid, specifically a bentonite polymer system. The research was concluded with a lower cuttings-transport rate for both fluids with increased yield point and plastic viscosity, at all inclination angles except vertical and near vertical condition. The described effect was higher for the invert emulsion mineral oil-based drilling fluid. Decreased yield point and plastic viscosity would then, together with higher flow rates, improve the hole cleaning for both fluid types. Furthermore they reported an observation of the same hole-cleaning performance for both fluid types at inclinations between 70 ° and 80 °, at low values of yield point and plastic viscosity. At higher values of yield point and plastic viscosity water-based drilling fluids provided better hole cleaning than the OBM. In contrast a study where oil-based drilling fluids protruded is given by Saasen and Løklingholm (2002). They defined the cause of the different behavior by the design of the drilling-fluid systems. Oil-based drilling fluids use a continuous oil phase and are viscosified with emulsified water and organophilic clay. The cuttings are not getting in contact with the emulsified water. Water-based drilling fluids have a brine phase which is viscosified by different polymers. Gelling in water-based drilling fluids is a primary cause of hole-cleaning problems. Drilling fluids with a low degree of shear thinning, give better hole-cleaning characteristics than fluids demonstrating a high degree of shear thinning. The authors addressed the better hole-cleaning capabilities of the oil-based

drilling fluids by claiming that in oil-based drilling fluids the effect of particle-particle gel forming is no longer existent, because the oil is inert on the particle surface. In accordance with Hareland, Azar, and Rampesad (1993), it is also mentioned that hole cleaning is improved with thinner fluids and that an optimum is reached when the yield stress and API gel strength values are as low as possible. On the other hand, precautions have to be made, because too low viscosities can lead to increased drill-string wear and barite sag (Omland et al. 2013) in the fluids.

In a comparative study by Hemphill and Larsen (1996), cutting-transport tests with water and base oil were done with a focus on the critical and sub critical flow rates. The critical flow rate was defined as the fluid velocity at which a cuttings bed begins to form. In addition a definition was made for angles greater than 35° from vertical. The so-called critical transport fluid velocity (CFTV) identifies the minimum fluid velocity required to maintain a continuously upward movement of the cuttings. The subcritical flow rates are fluid velocities lower than the critical flow rate which allows cuttings accumulation in the annulus. In their tests with water and base oil, water prevented the formation of a cuttings bed better at inclinations of 45°, 65°, and 85° from vertical. The base oil required ca. 20% to 25% more fluid velocity at the higher angles to achieve the critical flow rate than water. This phenomenon was most likely to be caused by the density difference.

Their work was concluded with the following points:

1. WBMs and OBMs will clean the inclined annulus similarly in well inclinations between 0° and 90°, under equivalent velocity and rheological conditions.
2. The fluid velocity is a key parameter in the cleaning of inclined annuli.
3. The mud weight is less important than the fluid velocity. In intermediate inclinations, the mud weight and its viscosities can affect cuttings transport.
4. Cuttings-bed instability is most pronounced at intermediate angles of inclination.

Under the presented conditions, water performed better than base oil when exposed to critical flow rates and subcritical flow. The critical fluid velocity is generally an important parameter to estimate the development of a cuttings bed (Ozbayoglu, Saasen, et al. 2010).

Hole cleaning is not only influenced by the type of drilling fluid, but also by drilling related properties like for example pipe eccentricity, drill-string rotation (Erge et al.

2015), and particle size and composition (Omland et al. 2005). Walker and Li (2000) investigated cuttings transport in coiled tubing operations with a focus on a broad variety of influencing parameters. During their experiments, particle size and fluid viscosity were tested, which included water, gel and multi-phase fluids. Additionally the pipe eccentricity was varied, what accounted to more than 700 tests with particle diameters of 0.15 mm, 0.762 mm and 7 mm. The critical deposition velocity was defined as the velocity in an inclined conduit that prevented the formation of stationary beds of solids in the lower part of the conduit. If the flow conditions were less than critical, solids fell out. The study highlighted the importance of the shear stress at the bed interface in terms of hole cleaning. Other influencing factors were therefore the flow regime, the geometry of hole/coiled tubing and eccentricity, which all were connected to the fluid viscosity. Another highlighted point was the difference between the hole-cleaning effect produced by the flow and the carrying capacity of the fluid. The authors pointed out the importance of the fluid viscosity. Low viscosity fluids in turbulent flow showed the best ability to pick up particles. This benefit of low viscosity was also seen by Ozbayoglu, Sorgun, et al. (2010). When they aimed to maximize the carrying capacity, they suggested a gel or multiphase system should be used. A complete cuttings removal could be achieved with extended periods of pumping time and not with the previous assumed "rule of thumb" of two times the hole volume. Fine particles were found to be the easiest to remove and particles with an average size of 0.76 mm showed the greatest difficulties.

Lower fluid viscosity for improved hole cleaning was also a conclusion by Adari et al. (2000). They used four different water-based fluids to execute an extensive experimental campaign on the influence of drilling-fluid rheology and flow rate to cuttings-bed erosion. One goal was to find the optimal combination of drilling-fluid rheology and flow rate. They concluded their work with the following points:

1. Cuttings-bed erosion increases with increased flow rate and turbulent flow
2. For a constant flow rate, lower cuttings bed height is achieved by higher n/K ratios, meaning enhanced cuttings removal by reduced viscosity
3. Higher deviated wells impede hole cleaning

The annular pressure loss (frictional pressure drop) was defined as a key parameter in hole cleaning by Saasen (1998) and Gavignet and Sobey (1989), and additionally found to have a high effect on other drilling parameters (Erge et al. 2015). The larger the frictional pressure drop, the better the hole cleaning became. Another highlighted point

was the effect of cuttings-bed consolidation. In this term the author included the effects of drill-pipe rotation and viscosity. With no drill-pipe rotation the flow in the narrow part of the annulus was reduced if the degree of shear thinning was increased. When, in the same time, the frictional pressure drop was kept constant the results of Zamora and Hanson (1991) suggested that hole cleaning was more difficult with shear thinning fluids. It was concluded that the drilling-fluid viscosity, as measured by standard methods, was not a major property affecting hole cleaning. Higher importance was given to the fluids ability to form gel structures within the cuttings bed. For oil-based drilling fluids, the gravity force was the dominant force keeping the cuttings in position at the surface of the cuttings bed. With high enough shear forces to lift a cuttings particle, it will be removed. For water-based drilling fluids the gelling is influenced by a combination of water, solid particles and polymers. Very complex inter-particle forces bind the particles between themselves in water and so to the cuttings bed. Important was also the effect of large molecular weight polymers which can form a gel structure. This gel structure can resist large strain. Saasen concluded with the following points:

1. The frictional pressure drop is the primary factor affecting hole cleaning.
2. Sufficiently high annular frictional pressure loss is important to obtain good hole cleaning.
3. Different types of drilling fluids create different degrees of consolidation of cuttings bed.
4. The importance of evaluating the degree and type of cuttings bed consolidation.

1.3 Drilling-Fluid Rheology

At some times during drilling the pumping process stops. This can be either planned breaks due to connecting additional drill pipes, or unplanned breaks due to problems during the operation. For this reason drilling fluids are designed to build up a structure while at rest. When the fluid flow stops the suspended cuttings will start to settle due to gravitational forces. The buildup of a fluid structure is slowing down or hindering this settlement and keeps the cuttings suspended in the fluid. When the pumping is resumed less energy is required to restart the fluid circulation and an accumulation of cuttings in the annulus is avoided. The technical term to describe the structure buildup over time is *thixotropy* (Barnes 1997, Mewis 1979, Mewis and Wagner 2009). The *yield stress* of a

fluid is another relevant parameter when characterizing the structure in a fluid, representing the shear stress at zero shear rate in a flow curve. Determining a value for the yield stress is dependent on the measuring method and the regression method (Møller, Mewis, and Bonn 2006, Barnes 1999, Maxey et al. 2008). Thixotropy and yield stress are two different phenomena, but often present in the same fluids. Tehrani (2007) pointed out the relevant rheological properties of drilling fluids, listing the high-shear viscosity, the yield stress and the gelling properties. The high-shear viscosity is affecting the frictional pressure drop while the drilling fluid flows in the drill pipe and the annulus and determines therefore the pump requirements. Hence, high pump rates are required for high pressure drops, at the same time inducing the risk of fractures in weak formations, with a possible result of mud loss downhole. The yield stress gives a value to the solids suspending capacity of the fluid which is particularly important when the fluid is stationary.

Having control on the viscosity and density of drilling fluids during drilling is an important part to assure safe and successful operations. In the field Fann 35 viscometers and mud balances for density measurements are the widely used measuring systems, following the API 13D/ISO standard 10414 (API 2010). The standard includes the measurement of the shear stresses at six different shear rates and the gel strength of the fluid after 10 s and 10 min of rest. Rheometers like the Anton Paar MCR enable more detailed measurements with self-selected amounts of measuring points. The complexity of drilling fluids opens the possibility to investigate viscoelastic properties. Important characteristics, such as yield stress, gel strength, linear viscoelastic range (LVER), or thixotropy can be measured and estimated accurately using rheometers. Schulz, Strauß, and Reich (2013) and Jachnik (2003) pointed out reasons for an extended investigation of drilling fluids with rheometers and show the advantages compared to Fann 35 viscometers. The importance of the low-shear viscosity is mentioned, as well as the gel strength. The 10 s gel strength value could be higher than the 10 min gel strength value, which could lead to negative thixotropy values due to altering. The work was concluded with an establishment of precise flow curves due to the increased number of measuring points. The cross-over point in amplitude-sweep tests was used to obtain a value for the yield stress. Bui et al. (2012) investigated linear viscoelastic properties of drilling fluids using an Anton Paar MCR 301 rheometer and conducting periodic oscillatory tests to evaluate gel strength and time and temperature dependence. Highlighted were the possibilities viscoelastic data can provide to drilling operations, and the lack of a model

to display the low-shear viscosity of the drilling fluids, including thixotropy and elastic effects. The viscoelastic data would help to model pressure peaks and pressure profiles in start-up circulation in offshore drilling. During amplitude tests with different frequencies, the LVER and the dynamic yield point were found to be higher leading to the conclusion that immediate resistance to breaking the gel strength of a drilling fluid is higher under fast deformation. In practical terms a slower increase of pump pressure to break the gel could reduce the pressure peak. The formation of gel structure was investigated with oscillatory time-sweep tests. It was found out that the structure builds up fast right after rest and sometimes reached stable values after longer times than recommended by API (30 minutes). Unfortunately the authors did not provide information about which fluids were tested, making it hard to compare results. More recently Bui and Tutuncu (2014) proposed cubic splines to model flow curves from data obtained by field viscometers. The cubic splines showed good match with any set of experimental data. This approach requires an increased number of equations and parameters, but is believed to be easily usable in the field, and to improve the accuracy of drilling-hydraulics modeling. Altindal et al. (2017) examined the viscoelastic properties of drilling fluids by comparing rheometer data with settling velocity experiments and mathematical modeling.

The structure buildup in drilling fluids was investigated by Maxey (2007), who studied effects of thixotropy and yield stress of water-based and oil-based drilling fluids with rheological measurements. An evaluation was made with various traditional and several recently proposed yield-stress models. Three different measurement systems were used, an Anton Paar MCR 301, a Rheometrics RFS-3 and an OFI-900 viscometer. The flow properties were found to be highly dependent on the measurement method, especially the influence of time and the mode, strain or stress controlled, were pointed out. While modeling, no single model predicted the behavior of a full test and the predicted yield stresses were sometimes far off the experimentally obtained values. Herzhaft, Ragouillaux, and Coussot (2006) showed a simple thixotropic and structural model to describe all the features of the rheological behavior of drilling fluids by using data from Fann 35 viscometers. The model is able to describe steady state and transient rheological properties of the drilling fluids without the need to define values for macroscopic characteristics, i.e. yield stress and gel building, in advance.

A study which compared effects of drilling-fluid rheological parameters on annular drill-cuttings buildup with a large scale flow loop was made by Becker, Azar, and Okrajni (1991). The investigated fluids were 15 bentonite/polymer water-based drilling fluids and the well inclinations were ranging from 30° to 70°. Viscosity showed its greatest influence at angles closer to vertical. Their analysis showed that at high inclinations the reduced cuttings volumes were more a result of flow-regime transitions than of variations in mud viscous forces. The particle concentration was correlated to various rheological parameters obtained by Fann V-G meters. The 6 rpm dial reading correlated best with the cuttings transport performance. Mud-shear stress at average annular shear rate, the 3 rpm dial reading, and the initial gel strength also correlated well. When the flow turned turbulent the drilling-fluid rheology did not show any noticeable effect on the cuttings concentration.

1.4 Objective

Field experience shows that oil-based and water-based drilling fluids clean the borehole differently, even when they show similar viscosity profiles. The reasons for this are not completely understood. Different explanations have been formulated on whether the reason is the fluid rheology, the resistance to bed erosion, both together, or something else. The aim of this study is to make a contribution to the understanding of the different behavior of oil-based and water-based drilling fluids and their ability to transport cuttings out of the borehole. The two main focusing points are defined as:

a) Rheological determination of oil-based and water-based drilling-fluid properties,

b) A comparative experimental study of cuttings transport to test oil-based and water-based drilling fluids with similar viscosity profiles and densities.

The research project combines the rheological investigation of drilling fluids with flow-loop experiments. Such experimental investigations comparing drilling fluids with a similar viscosity profile under identical and controlled conditions are very limited in the literature. Advanced measuring with rheometers gives a deep insight into the physio-chemical properties of the drilling fluids and can help to understand behavioral

differences. The low-shear viscosity is rarely investigated in hole-cleaning studies in the present literature, but is most likely a relevant parameter in explaining the differences. The combination of a field relevant scaled flow loop and rheological measurements will increase the understanding of fluid properties and hole-cleaning behavior, and enable the design of advanced and efficient fluid systems with minimum environmental impacts. Efficient drilling is one of the main challenges in the drilling industry and can be achieved with lowering operational time and/or costs. Drilling faster can be realized with better wellbore cleaning and an improved borehole condition. This will further reduce drag and torque, non-productive time, and the risk of stuck pipe. In a broader perspective, increased understanding of drilling-fluid properties will help to enhance the development of new technologies related to for example automated drilling (Saasen et al. 2009, Omland et al. 2007) and flow modeling and hole-cleaning prediction (Zhang et al. 2015, Hashemian et al. 2014).

1.5 Research Method

To investigate the different behavior of oil-based and water-based drilling fluids and to research the influence of the low shear rheology, two experimental campaigns are being performed:

    a) Rheological characterization and analysis of drilling fluids with a Fann 35 viscometer and an Anton Paar rheometer.

    b) Controlled cuttings-transport experiments with a simplified sand bed in a flow loop.

For the experiments three oil based and one water-based drilling fluid will be used. The oil-based fluids are water-in-oil emulsions with a yield stress (named OBM A, B, and C), containing barite, CaCl2, bentonite, lime, emulsifier, and a fluid loss agent. The water-based fluid is a KCl fluid, viscosified with xanthan gum. Other additives are glycol, polyanionic cellulose, starch, soda ash and barite. The fluids will be adjusted to have a similar viscosity profile in the relevant shear-rate range of the flow-loop experiments. A detailed description of the fluid composition and properties is presented in chapter 2.2 and Table 3.

The rheological characterization includes the measurement of flow curves, amplitude-sweep curves, temperature dependence, and thixotropy. These experiments will reveal the material properties and the flow behavior at very low flow conditions and shear rates.

To relate to drilling operations in the field, relevant wall-shear rates ($\dot{\gamma}_w$) are calculated by using equation (1), where U represents the fluid velocity, D the borehole diameter, and d the drill-pipe diameter (Saasen et al. 2004). The equation can be used for an estimation, but the results will somewhat differ from the correct values as the non-Newtonian behavior of the fluids and the drill-string rotation are not taken into account. The calculations are made for different realistic borehole diameters, drill-pipe diameters and maximum and minimum pump rates.

$$\dot{\gamma}_w = \frac{12\,U}{D - d} \tag{1}$$

Table 1 presents the relevant high shear rates for three typical borehole sizes. The shear rates are graphically displayed in Figure 1 together with the Fann 35 measurements of the KCl fluid and the OBM B. For example the orange line is calculated for a borehole size of 8,5". At a pump rate of 2000 lpm the highest shear rate is 247 1/s. The area left of the orange line in Figure 1 represents than the relevant shear rate in the flow-loop experiments. Water-based and oil-based drilling fluids will never be 100% matching, as water-based drilling fluids usually show a flatter trend.

As can be seen in Figure 1, the estimated shear rates in the borehole are in the lower shear-rate region. The shear rate in the cuttings bed, when initiating movement between cuttings in a consolidated cuttings bed will be even lower.

Table 1. Relevant high and low shear rates for typical borehole sizes.

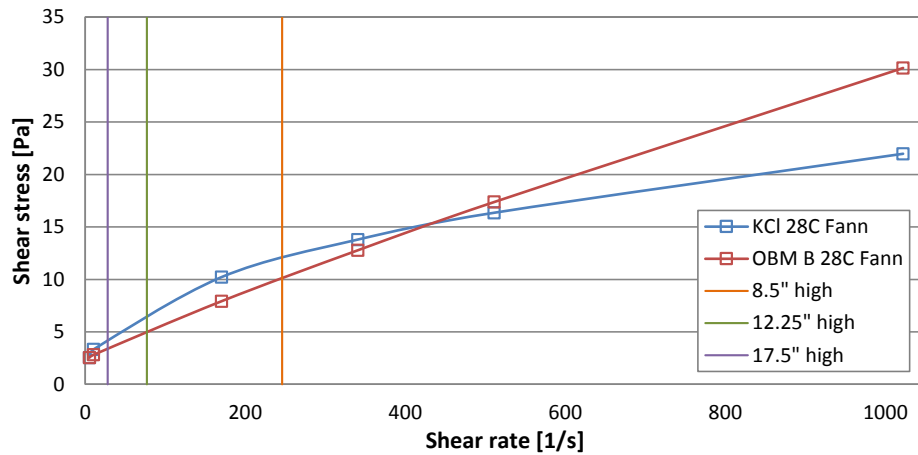| Hole Diameter ["]	DP Diameter ["]	High Pump Rate [lpm]	Low Pump Rate [lpm]	High Shear Rate [1/s]
17,5	5,5	6000	4500	28
12,25	5,5	4000	3400	77
8,5	5,5	2000	1000	247

Figure 1. Fann 35 measurements of the KCl fluid and OBM B at 28 °C together with typical high and low shear rates for typical borehole sizes.

A custom build flow loop (Figure 2) is used to conduct cuttings-transport experiments with the same drilling fluids under controlled conditions. The flow-loop setup is a result of the work by Taghipour (2014) who originally investigated hole cleaning in non-circular wellbores. The 10 m long test section is equipped with concrete segments, which represent the borehole, and a fully eccentric inner rotating drill string, which can swirl freely in the annulus. One side of the drill string is connected to the drive motor via a cardan shaft, and the other side is free. Sand, together with the drilling fluid, is pumped into the test section to build up a sand bed. When the sand bed is established the experiment is started. Different fluid velocities and/or drill-string rotations are the test parameters. The fluid-sand mixture exits the test section and flows into a vacuum-rotation filter system (VRF) where the fluid is separated from the sand. The VRF also works as a storage for the drilling fluid.
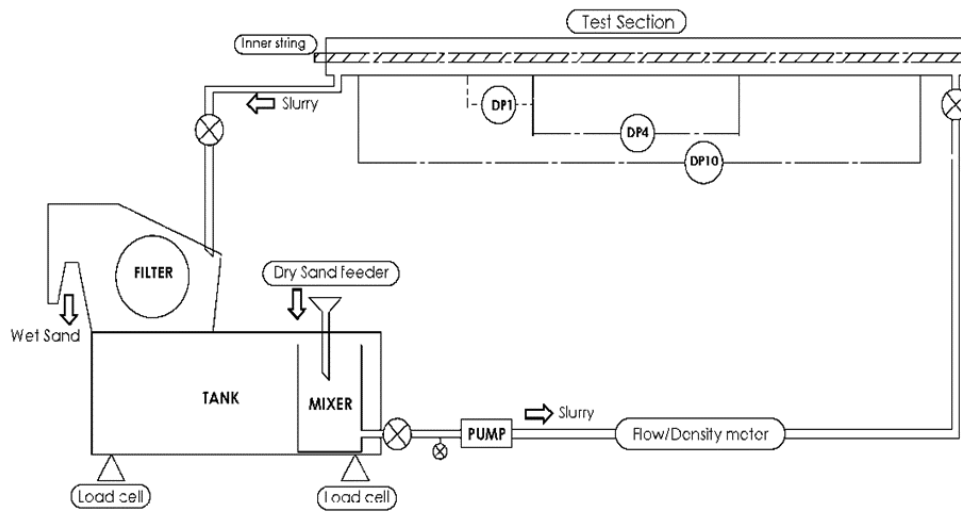
Figure 2. Schematic drawing of the flow loop set up for controlled cuttings-transport experiments.

2. Comparative Rheological Investigations

2.1 Measurement Systems and Procedures

Two measurement systems were used during the experiments to characterize drilling fluids. A Fann 35 viscometer was used to measure the viscosity and an Anton Paar MRC rheometer to measure other rheological parameters.

2.1.1   Fann 35 Viscometer

In the oil industry the Fann 35 viscometer is the measuring device referred to in the standards API 13D and ISO 10414 (International-Standard 2011). The device was primarily used to keep track of the fluid condition during preparation of the fluids for the flow-loop experiments, and the actual execution of the flow-loop experiments. It is based on a Couette rotational viscometer and the fluids are tested in the annular space between the measuring bob and an outer rotating cylinder. The outer cylinder exerts a viscous drag force to the fluid. The creating torque is measured with the bob and transmitted to a dial reading via a torsional spring. The Fann 35 is operated with six different rotational speeds (600 rpm, 300 rpm, 200 rpm, 100 rpm, 6 rpm, 3 rpm), based on the API recommended practice (API 2010). The measurement procedure starts with a pre-shear period at 600 rpm, afterwards the measurements are taken with decreasing rotational speeds when the dial reading is stable. The final part of the measurement consists of gel-strength measurements after 10 s and 10 min of rest. Measurements were taken at temperatures of 28 °C and 50 °C with a R1 rotor sleeve, a B1 bob, and a F1 torsion spring configuration. 28 °C was the operational temperature of the flow loop and 50 °C the recommended temperature of the standard.

2.1.2   Anton Paar Rheometer

The Anton Paar MCR 102 and 302 rheometers used in this study provide high accuracy due to high precision air bearings and a powerful, synchronous EC motor drive. For the measuring system a CC27 bob cup set-up (Figure 3) was chosen to minimize evaporation effects at measurements with elevated temperatures. In contrast to Fann 35 viscometers the rheometer enables to measure in the low and very low shear-rate range, and can be operated in oscillation mode. Additionally measurements can either be shear stress or shear rate controlled. A software package gives full control over test settings and provides analysis options. To ensure homogeneous fluids and similar test conditions in the laboratory over several days of testing, a pre-treatment procedure was established.

Every morning the fluid batch was mixed in a Waring blender at about 6000 rpm for 10 minutes, followed by a rest time of 1 hour. From this fluid batch smaller samples were used for the experiments. After each conducted measurement the sample was replaced with an unused one. The temperature was controlled and adjusted by a Peltier element.
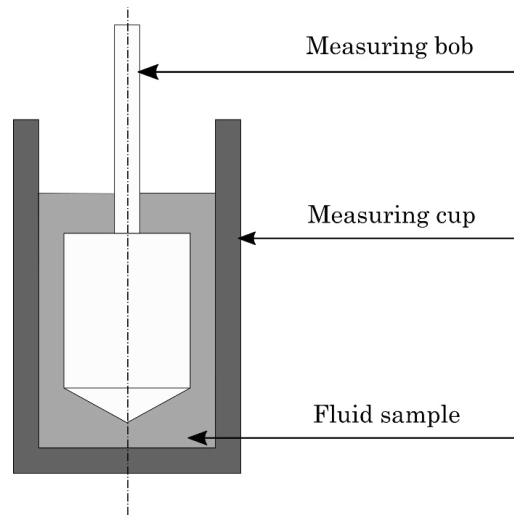

Figure 3. CC27 set-up used in Anton Paar rheometer experiments.

2.1.3 Rheometer Experiments

To determine viscoelastic properties of drilling fluids different experiments were conducted. All tests were done at temperatures of 28 °C and 50 °C, and some tests additionally at 10 °C. The selection of the different tests described in the forthcoming paragraphs was mainly based on previous studies (Torsvik et al. 2014, Ytrehus et al. 2014).

Flow curves

Flow curves are measured by either controlled shear rate or controlled shear stress and show the viscosity function of the material. Simple flow curves were conducted with linear increasing shear rate from 1 – 1200 1/s and 120 measuring points.

To enhance understanding of the low shear behavior, low shear flow curves in the range of 0.01 – 100 1/s and 0.001 – 100 1/s were done with a logarithmic increase and constant measuring-point duration of 2 s, which resulted in 80 s of total measurement time. Additionally, the 0.001 – 100 1/s experiments were performed with a decreasing

measuring-point duration from 30 s to 2 s to avoid transient effects. The total measuring time accounted to 640 s.

Amplitude-sweep tests

Amplitude-sweep tests are oscillatory tests with a constant frequency and increasing amplitude. The frequency was set to 10 1/s and the amplitude increased from 0.001 to 100 % strain with a slope of 5 measuring points per decimal, accounting to 26 measuring points. The outcome of the measurements are curves of the storage modulus (G') and the loss modulus (G''), characterizing the materials elastic, viscous, or viscoelastic behavior. If G' > G'', the elastic behavior dominates over the viscous behavior and the sample shows a solid like character. The relation of the storage to the loss moduli then gives a measure of the stiffness of the material. In the opposite case where G'' > G', the viscous behavior is dominating and the sample acts liquid like. If the curves are crossing each other (G' = G''), the point is called *flow point*. The ratio between G'' and G' is called the loss factor tan δ. When tan δ > 1, the sample shows a more viscous behaviour, and respectively a more elastic behaviour when tan δ < 1. The length of the linear viscoelastic range (LVER) indicates the minimum strain to initiate breakage of the inner structure and determines the strain value for 3-interval-thixotropy tests.

3-Interval-thixotropy-tests

3-Interval-thixotropy-tests help to understand the structure-rebuilding character of materials. The tests are performed in 3 steps. During the rest interval the sample is oscillated at a constant frequency and a strain value inside the LVER, as obtained from an amplitude sweep. In the load interval, the sample is sheared at a constant shear rate to break the internal structure. During the terminatory recovery interval, the sample is again oscillated at the same parameters from the rest interval, to investigate the structure rebuilding character of the sample. Test parameters were selected as presented in Table 2.

Temperature-sweep tests

Temperature-sweep experiments show the viscosity dependency of the temperature. The samples were sheared at a constant shear rate of 100 1/s while the temperature was increased from 5 – 50 °C with a slope of 1 K/min.

Shear-stress sweep tests

Experiments with controlled shear stress are used to determine the yield stress of a material. During the tests the shear stress was increased logarithmically from 0.1 – 1000 Pa, with 200 measuring points and a measuring-point duration of 1 s. The yield stress can be estimated where the strain-stress curve deflects from linearity in a shear strain vs. shear-stress diagram.

Table 2. Experimental settings for 3-interval-thixotropy-tests, γ represents the strain and γ̇ the shear rate.

	Rest interval	**Load interval**	**Recovery interval**
1	γ = 0.1 %	$\dot{\gamma} = 10\ 1/s$	γ = 0.1 %
	10 measuring points, 20 s measuring point duration, 200 s measuring time, f = 10 1/s	10 measuring points, 0.1 s measuring point duration, 1 s measuring time	10000 measuring points, 10 s measuring point duration, 100.000 s measuring time, f=10 1/s
2	γ = 0.1 %	$\dot{\gamma} = 1000\ 1/s$	γ = 0.1 %
	10 measuring points, 20 s measuring point duration, 200 s measuring time, f = 10 1/s	10 measuring points, 0.1 s measuring point duration, 1 s measuring time	10000 measuring points, 10 s measuring point duration, 100.000 s measuring time, f = 10 1/s

2.1.4 Definition of Yield Stress and Yield Point

The *yield stress* is an important parameter for structured fluids like drilling fluids (Barnes 1999, Maxey 2007, Boisly et al. 2014). Its value is dependent on the measuring method and/or the regression method and not a material constant (Dinkgreve et al. 2016). Different methods to determine a value are widely discussed in the literature (Cheng 1986, Power and Zamora 2003, Møller, Mewis, and Bonn 2006). In a flow curve, the yield stress is the stress value at zero shear rate. As it is not possible to measure at zero shear rates, a typical method is to measure flow curves and extrapolate to a shear rate of 0 1/s. Modern rheometers allow to measure at very low shear rates which makes the procedure more precise, and also include other yield-stress measurement options, such as amplitude sweeps, or shear-stress sweeps.

In the drilling industry the *yield point* refers to a value obtained by calculations based on Fann 35 viscometer measurements using two high shear values, following the API (2010).

2.2 Drilling Fluid Composition

During the study 4 different fluids were used, one water-based fluid called KCl fluid, and three oil-based fluids, called OBM A, OBM B, and OBM C. The three OBMs are the commercial *Versatec* fluids by MI-Swaco. The components are shown in Table 3 together with the density and oil-water ratio. All fluids are actual drilling fluids used in drilling operations. After use in the field they were cleaned, reconditioned and shipped to the research facilities of NTNU and SINTEF in Trondheim. Adjustments to the initial *Versatec* fluid had to be made to enable optimal handling in the separation machine and with the mud pump during flow-loop experiments. The viscosity was too high to separate the sand particles from the fluid with the pre-assembled mesh in the separation machine, also the pump could not pump the fluid. A change in mesh size and dilution from an initial density of 1.37 g/cm^3 with the corresponding base oil EDC 95-11 were chosen as a solution for the problem and resulted in OBM A and OBM B. The problems with the separation machine and the dilution of the fluids resulted in an altered oil-water ratio. Another try was made to conduct experiments with a higher viscosity and density fluid. After finishing the experiments with OBM B, Bentone 128 was added to OBM B and resulted in OBM C. The density was not highly affected by the Bentone 128, but the viscosity increased.

The KCl fluid has the commercial name *Glydril* and was initially planned to match OBM C. During preparation of the flow-loop experiments with the KCl fluid, the fluid was not moved enough in the tank, and heavy particles started to settle on the bottom of the fluid tank. Additionally the fluid level decreased to an insufficient small amount to conduct experiments, due to evaporation. It was decided to add water and xanthan gum in amounts to match OBM B, which was successfully accomplished.

It is not possible to design oil-based and water-based drilling fluids with exactly the same viscosity profile, but it is possible to get quite close. For the comparative hole cleaning experiments of the project, the OBM B and the KCl fluid were chosen. Figure 4 shows their flow curves measured with a Fann 35 viscometer. Both fluids show a similar

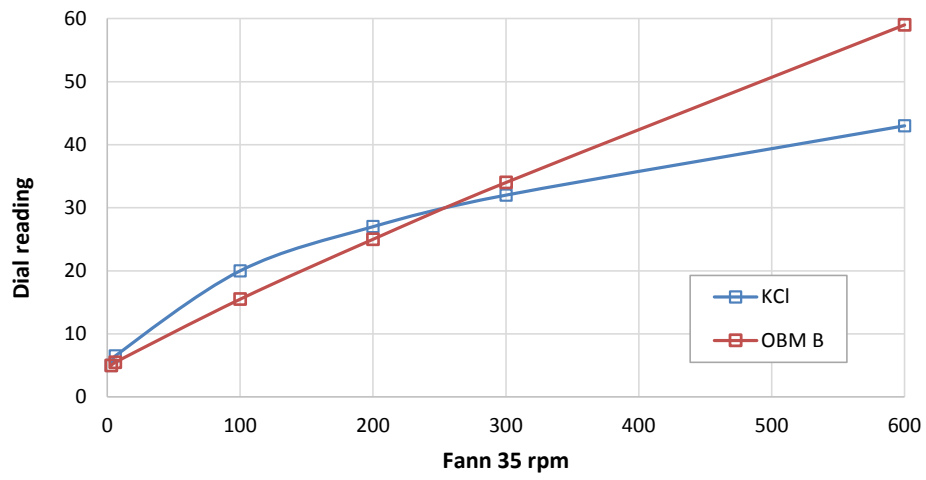trend in a shear-rate range between 0 1/s and about 500 1/s (0 to ca. 300 rpm in a Fann 35 viscometer).

Figure 4. Fann 35 measurements for OBM B and the KCl fluid for 28 °C.

Table 3. Components and properties of OBM A, OBM B, OBM C, and the KCl fluid.

	OBM A	OBM B	OBM C	KCl
Components	Base oil EDC 95-11	Base oil EDC 95-11	Base oil EDC 95-11	Fresh water
	Barite	Barite	Barite	KCl
	Salt (CaCl$_2$)	Salt (CaCl$_2$)	Salt (CaCl$_2$)	Glycol
	Organophillic clay (Bentonite)	Organophillic clay (Bentonite)	Organophillic clay (Bentonite)	Xanthan gum
				Polyanionic cellulose
	Lime (Ca(OH)$_2$)	Lime (Ca(OH)$_2$)	Lime (Ca(OH)$_2$)	Starch
	Emulsifier	Emulsifier	Emulsifier	Soda ash
	Fluid loss agent	Fluid loss agent	Fluid loss agent	Barite
			Bentone 128	
Oil-water ratio	Initially 80/20	Initially 80/20	95/5	Not applicable
Density [g/cm^3]	1.11	1.26	1.27	1.19
Regression parameters 28 °C	τ_0 2.9124	3.271	9.9282	-
	K 0.040533	0.80284	0.14637	0.42975
	n 0.88813	0.10001	0.7983	1.0362
	R^2 0.99975	0.99849	0.99947	0.99596
Regression parameters 50 °C	τ_0 1.8705	2.2522	4.9226	-
	K 0.037147	0.054883	0.13747	0.49331
	n 0.84388	0.1999	0.75246	0.55456
	R^2 0.99935	0.99643	0.99984	0.99426

Table 4. Fann 35 dial readings for OBM A, OBM B, OBM C, the KCl fluid, and ES values for the oil based fluids.

Fluid	Temperature [°C]	Fann 35 rotational speeds [rpm]								ES
		600	300	200	100	6	3	10 s	10 min	
OBM A	28	40	23	17	11	3.5	3	-	-	Not measured
	50	-	-	-	-	-	-	-	-	
OBM B	28	59	34	25	15.5	5.5	5	-	-	800-900
	50	34	19	14	9	3	2	3	5	
OBM C	28	94	56	41	26	8	7	11	22	1500-
	50	64	39	29	19	6.5	6	9	19	1900
KCl	28	43	32	27	20	6.5	5	5	6	Not
	50	36	27	23	17	5	4	4	5	applicable

2.3 Results of the Rheological Characterization

2.3.1 Preconditioning Procedures for the Rheological Characterization of Oil-Based and Water-Based Drilling Fluids

The time and stress dependence of thixotropic fluids is characterized by decomposition and regeneration of the samples structure. The measurement outcome will depend on which phase the sample`s structure is in at the start of the experiment. Therefore it is important to have a consistent pretreatment procedure when conducting rheological measurements on such fluids. The shear history can affect the results, and influence reproducibility.

Flow curves and amplitude-sweep tests were conducted using a Fann 35 viscometer and an Anton Paar MCR 302 rheometer with various pretreatment procedures to quantify the effects of pre-shearing, no pre-shearing, and rest on the results. The first set of experiments was done without pre-shear, and different waiting times of 1 hr, 2 hr, 4, hr, 6 hr, 8 hr, and 24 hr. The second set of experiments included a 10 min pre-shear interval (600 rpm, 1022 1/s) after the waiting time, and prior to the measurement. All experiments were done at temperatures of 28 °C and 50 °C with OBM C (Table 5).
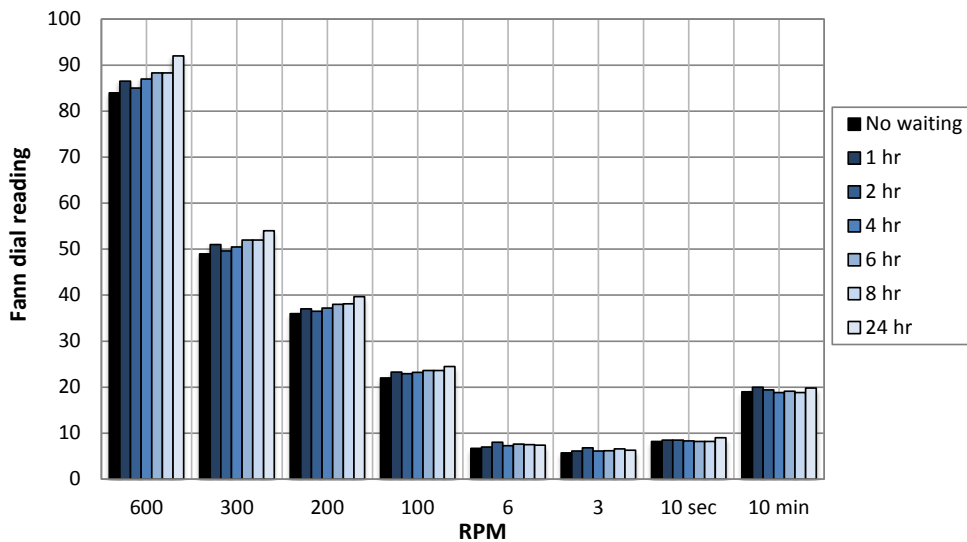
Figure 5. Fann 35 dial readings of OBM C taken without pre-shear at a temperature of 28 °C. Waiting times as indicated, the lighter the color, the longer the waiting time.

Table 5. Test matrix for preconditioning experiments

Set	Procedure
1 – No pre-shear	Waiting time of 1 h, 2h, 4 h, 6 h, 8 h, 24 h
	No pre-shear
2 – Pre-shear	Waiting time of 1 h, 2h, 4 h, 6 h, 8 h, 24 h
	10 min pre-shear (600 rpm, 1022 1/s)

Figure 5 shows a plot of Fann 35 dial readings without pre-shear at 28 °C. The difference in dial-reading values for 600 rpm was a 9 % increase from no waiting time to 24 hr waiting time, and between 2 % and 10 % for 300 rpm to 100 rpm. The values for 6 rpm and 3 rpm appeared stable and not effected by the waiting time. This is also true for the 10 s and 10 min gel-strength readings. The fluid has been sheared significantly before these last four measurements which could be an explanation for the stable values.

A flatter trend is visible for experiments with pre-shear, as shown in Figure 6. The maximum increase in dial reading values was 4 % (at 600 rpm).
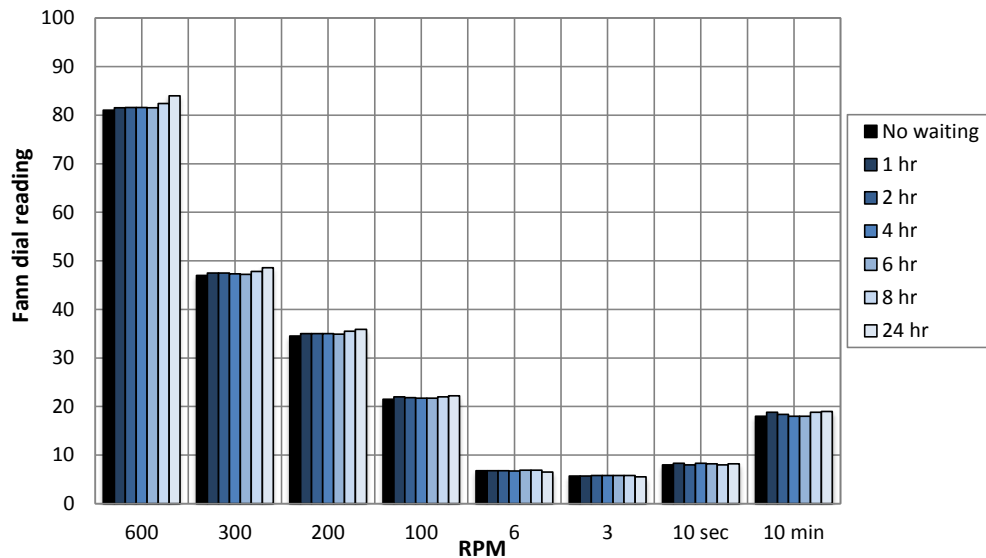
Figure 6. Fann 35 dial readings of OBM C taken with pre-shear at a temperature of 28 °C. Waiting times as indicated, the lighter the color, the longer the waiting time.

When comparing Figure 5 and Figure 6, it is clear that pre-shearing leads to more reproducible results, except for the measurements with the longest waiting period of 24

hr. The same trends were seen in the 50 °C measurements and also in the flow curves conducted with the Anton Paar rheometer, which are not presented here but can be seen in paper 1.
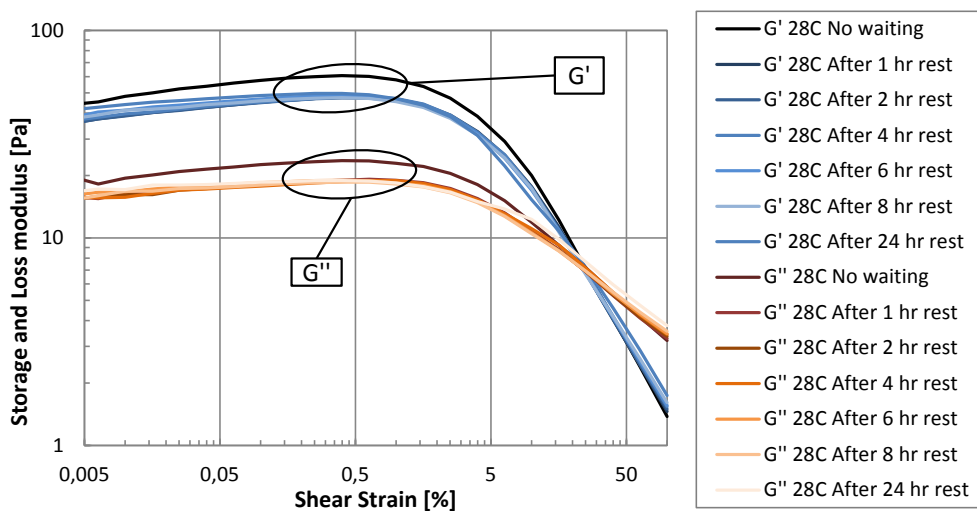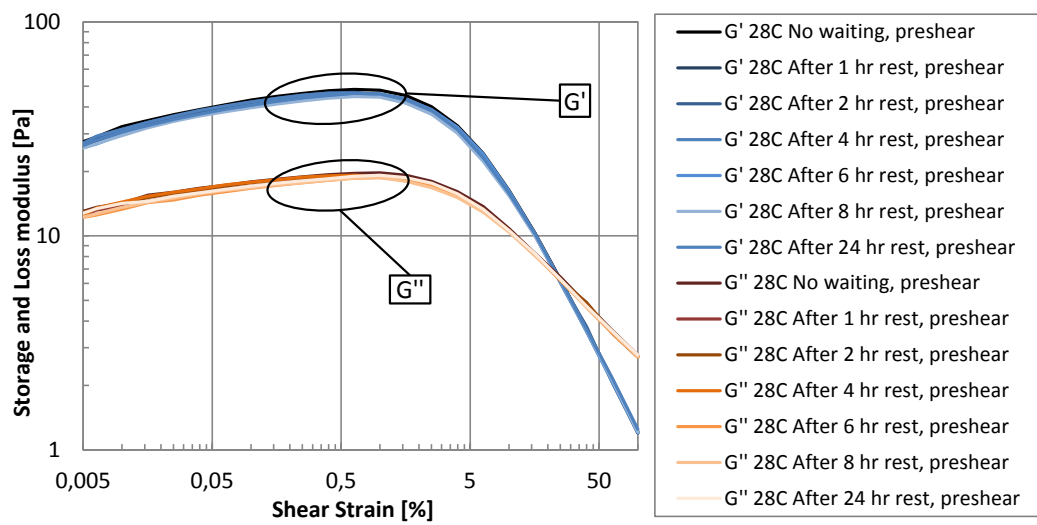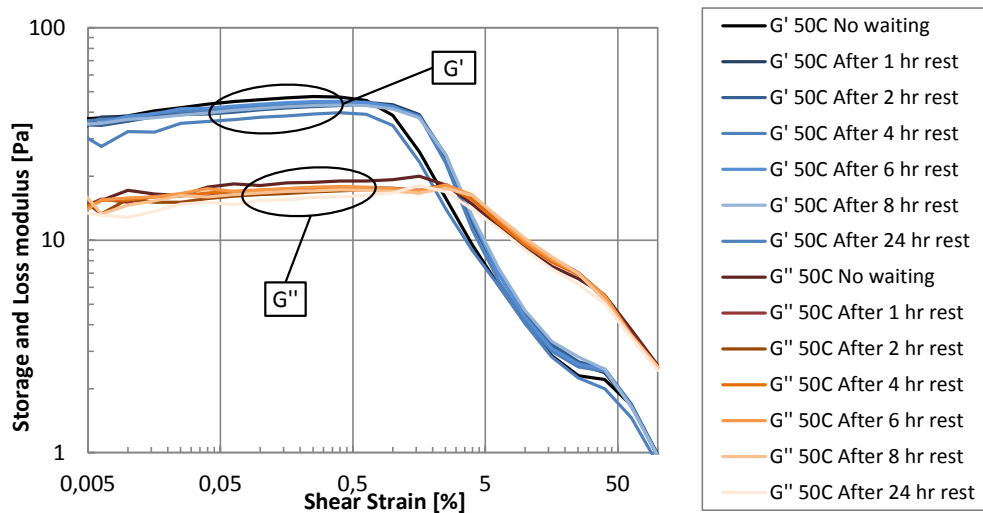

Figure 7. Amplitude sweeps without pre-shear of OBM C at 28 °C. Waiting times as indicated, the lighter the color, the longer the waiting time, measured with Anton Paar MCR 302.

Figure 7 shows amplitude sweeps without pre-shear and Figure 8 shows amplitude sweeps with pre-shear for 28 °C. When comparing the two graphs it is observed that the reproducibility was improved by pre-shearing. The curves in Figure 8 are much closer to each other than the curves in Figure 7. The cross-over point did not change, but the end of the LVER was moved to higher strain values, meaning that the fluid was able to tolerate more strain before the structure started to break down. Figure 9 and Figure 10 show the amplitude-sweep curves for a temperature of 50 °C. Here pre-shearing also resulted in more identical curves than without pre-shearing, indicating an increased reproducibility. At the higher temperature the cross-over point was shifted to ca 20 % higher strain values, and the LVER increased as well.

Additional graphs can be found in appendix A.1.

Figure 8. Amplitude sweeps with pre-shear of OBM C at 28 °C. Waiting times as indicated, the lighter the color, the longer the waiting time, measured with Anton Paar MCR 302.

Figure 9. Amplitude sweeps without pre-shear of OBM C at 50 °C. Waiting times as indicated, the lighter the color, the longer the waiting time, measured with Anton Paar MCR 302.
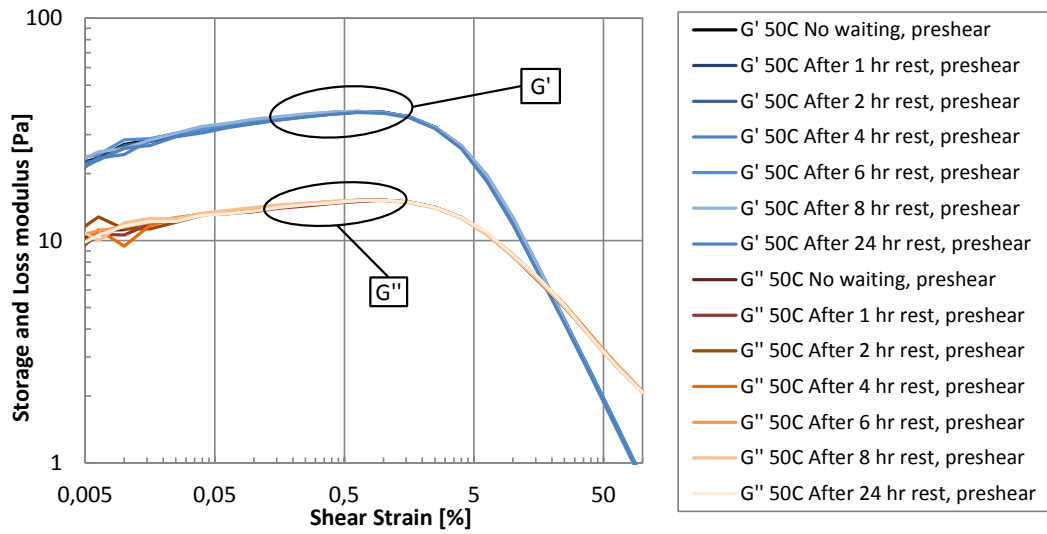
Figure 10. Amplitude sweeps with pre-shear of OBM C at 50 °C. Waiting times as indicated, the lighter the color, the longer the waiting time, measured with Anton Paar MCR 302.

A similar investigation of the pre-conditioning effects like those of the oil-based fluid was performed with water-based fluids containing laponite. The fluids were aqueous suspensions of laponite with varying concentrations of xanthan gum and NaCl. The aim of the investigation was to quantify the impact of pre-shear and waiting time, and how this is affected by temperature, salinity and the addition of polymer. The composition is shown in Table 6. Pre-shearing was done for 2 min at a shear rate of 1020 1/s. The pre-shearing time was reduced from 10 min to 2 min, because in the experiments with OBM C it was seen that 2 min is enough. For experiments with waiting time, a period of 24 hr was chosen.

Table 6. Fluid composition of laponite suspensions.

Fluid	1		2		
Component	a	b	a	b	c
Water	Deionized water				
Laponite RD [wt %]	1.5	1.5	1.5	1.5	1.5
Xanthan Gum [wt %]	0	0	0.1	0.1	0.1
NaCl [g/l]	0	0.6	0	0.6	12
NaOH [mmol/l]	0.1	0.1	0.1	0.1	0.1
Biocide	0	0	0.1	0.1	0.1
pH value	10.07	10.31	10.51	10.21	9.31
Conductivity [mS]	0.576	1.525	0.546	1.382	17.7

The Fann 35 results showed that pre-shearing for fluids without salt content was not sufficient to reproduce the values obtained without waiting time. Pre-shearing was more efficient for a NaCl concentration of 0.6 g/l (Figure 11). For experiments without waiting time the pre-shear treatment did not show much effect. The observations from the Fann 35 viscometer could be confirmed with the Anton Paar rheometer. Compared to the corresponding Fann 35 measurement an increased slope could be seen after exceeding a shear rate of 450 1/s (black circle in Figure 12) in the Anton Paar measurement. A likely explanation for this increase could be Taylor instabilities. Taylor instabilities are vortices formed in rotating Taylor-Couette flow (Taylor 1923) when the critical Taylor number (Ta_{crit}) is exceeded. They are caused by an exchange of stabilities and the result is a stable secondary flow pattern. The Taylor number was calculated with equation (2), where ω is the angular velocity, a the radius of the outer cylinder, b the radius of the inner cylinder, and v the kinematic viscosity. The Taylor number accounted to 1322, which is slightly below the critical Taylor number (White 2006).

$$Ta = \frac{\omega^2 b(a-b)^3}{v^2} = Ta_{crit} \approx 1700 \qquad (2)$$
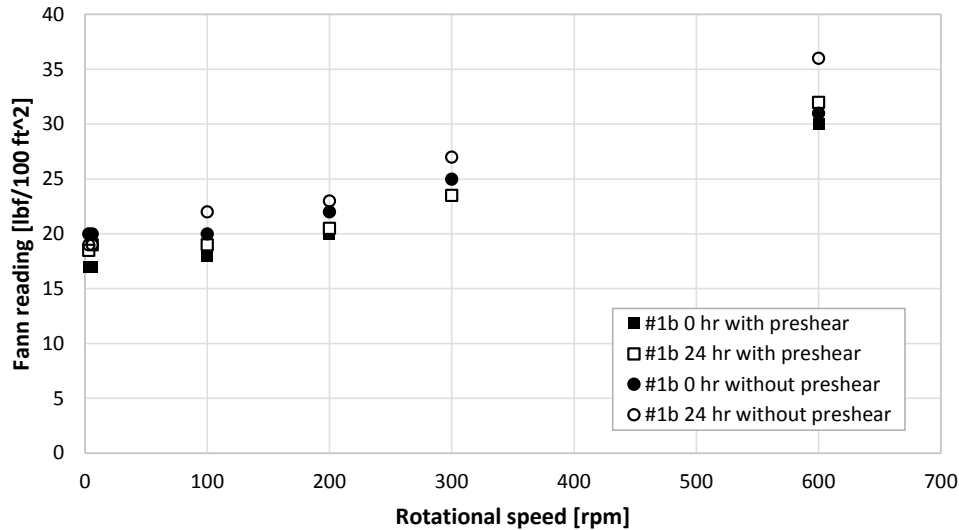
Figure 11. Fann 35 measurements for the laponite suspension #1b at 24 °C.
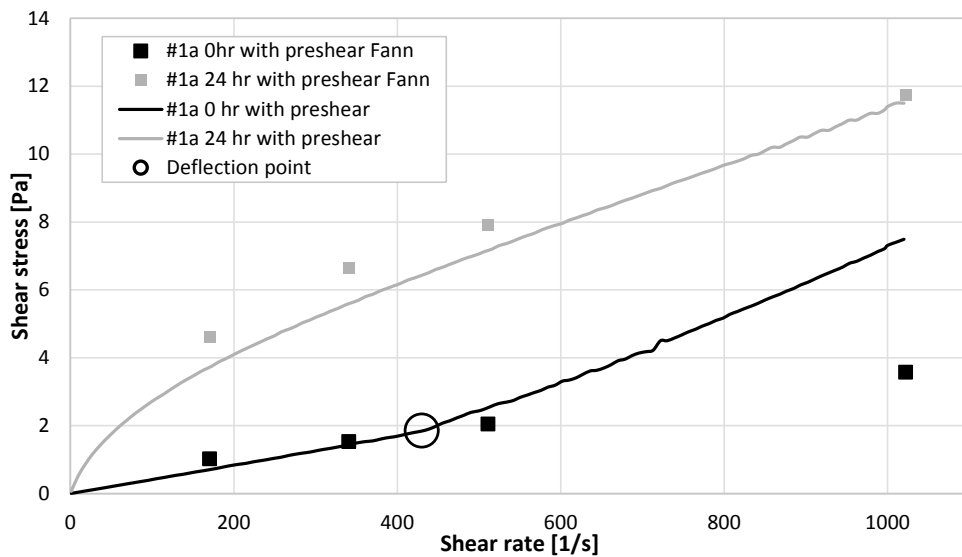
Figure 12. Comparison of Fann 35 and Anton Paar measurements for laponite suspension #1a at 24 °C (the dots represent the Fann 35 measurements and the lines the rheometer measurements).

The results from these experiments show the importance of consequent pretreatment of thixotropic fluids to obtain reproducible results. The effects of pre-shear and/or waiting time should ideally be tested for each fluid, but the results are expected to be valid for most oil-based drilling fluids. When testing viscoelastic properties, pre-shearing should be avoided, because the structure of the fluid will otherwise be destroyed. The viscoelastic properties were found to be most sensitive to pre-shear, especially at higher temperatures.

Additional graphs can be found in appendix B.1.

2.3.2 Flow Curves

The flow curves presented in Figure 13 were taken with the Anton Paar rheometer. All fluids showed a non-Newtonian trend and the OBMs showed yield stresses. The KCl fluid followed a Power-law trend, although a very small yield stress was visible. This yield stress is considered to have no practical relevance. The OBMs followed Herschel-Bulkley trends. All oil-based fluids exhibited lower shear stresses for 50 °C than for 28 °C. The details in the figure show the flow curves in a shear-rate range from 0 1/s to 100 1/s and the crossing point with the y-axis.
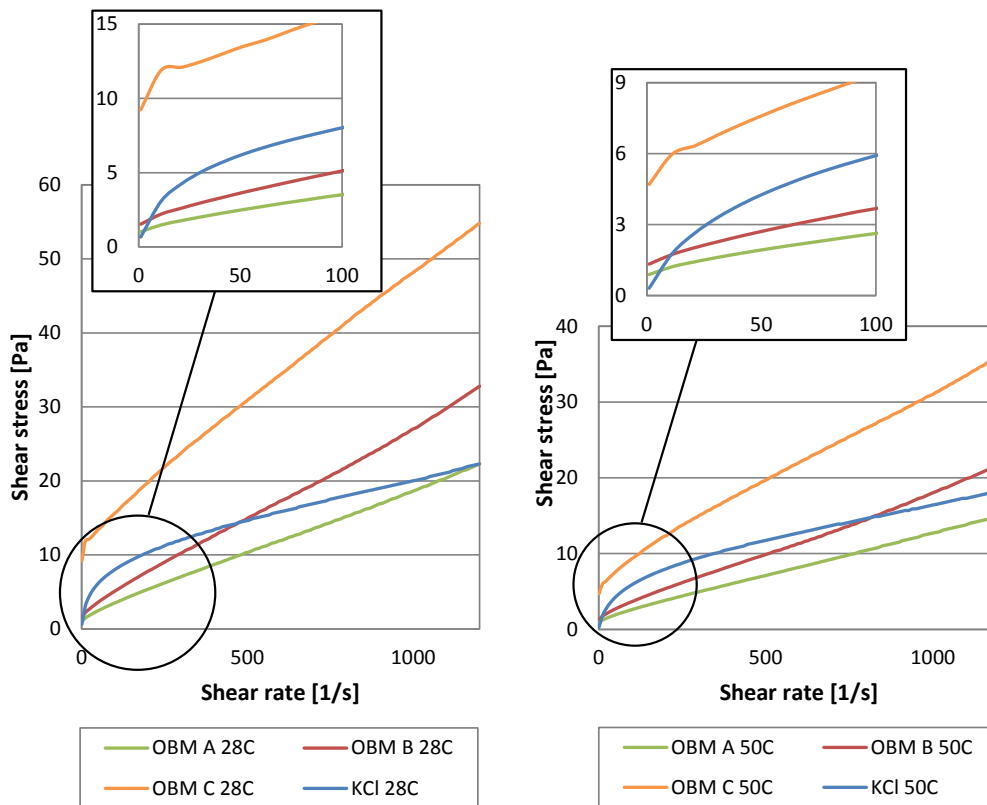
Figure 13. Flow curves measured with the Anton Paar rheometer for the OBMs A, B, C and the KCl fluid at 28 °C and 50 °C, measured with Anton Paar MCR 302.

When measuring flow curves with the Fann 35 a deviation from the rheometer results was seen. The Fann 35 curves showed lower shear-stress values than the Anton Paar

curves for the same shear rates. To investigate the underlying reason, a Fann 35 measurement was imitated with an Anton Paar rheometer. A 10 min pre-shear interval at 1022 1/s (600 rpm in Fann 35) was followed by measurements of shear stress at the corresponding Fann 35 rotational speed order of $600 - 300 - 200 - 100 - 6 - 3$ rpm. The plots are presented in Figure 14. The imitated Fann readings taken with the rheometer corresponded well with the initial Fann 35 measurements. The deviation between the solid and the dotted line is most likely caused by the differing measuring direction. The Anton Paar flow curves were done with increasing shear rate, whereas the Fann 35 measurements are done with decreasing shear rates. A shear history is induced into the fluid when measuring with a Fann 35 viscometer, resulting in lower shear-stress values, especially at lower shear rates. These findings correspond well with the results of the preconditioning study, where the pre-sheared samples showed lower shear-stress values compared to the not sheared samples.
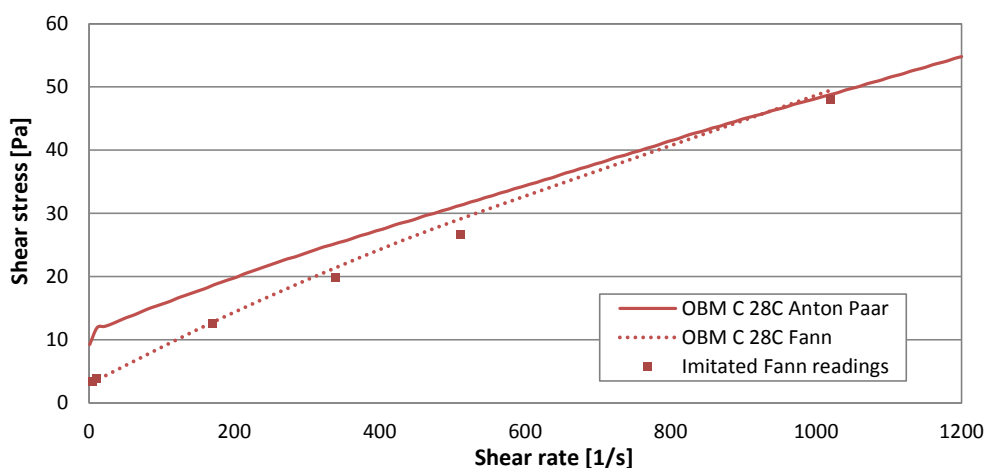
Figure 14. Comparison of OBM C flow curves measured with Fann 35 and Anton Paar, and imitated Fann 35 readings in the rheometer.

Figure 15 displays low shear-rate flow curves for the OBMs at 28 °C and 50 °C. At very low shear rates, just above 0.001 1/s, shear thickening tendencies are visible. Similar peaks also occurred after redoing the experiments and starting from a higher shear rate of 0.01 1/s (Figure 16). The reappearance of these peeks indicates rather a transitional effect than an increase in viscosity. A possible explanation could be the self-arrangement of water droplets in certain patterns, depending on the flow conditions in the dispersion. In the static case Brownian forces may reposition the water droplets of the emulsion to

reach a state where the droplets have, on average, the furthest possible distance to each other (Figure 17). This would result in a crystal like structure and high viscosity. Between 0.05 1/s and 0.2 1/s, a plateau like area is dividing the curves into two sections. Such behavior was also seen by Herzhaft et al. (2002), who found a transition between Newtonian behavior at low shear rates and non-Newtonian behavior at higher shear rates.
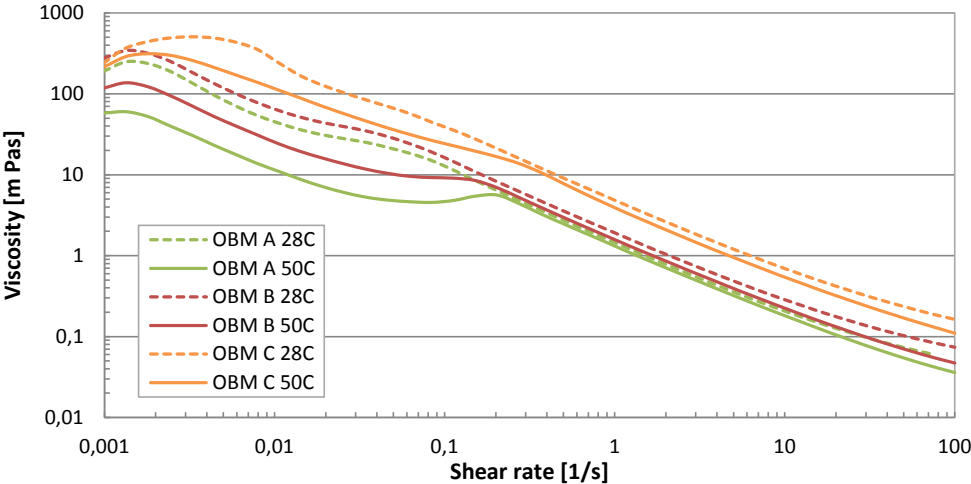
Figure 15. Low shear-rate flow curves in a shear-rate range of 0.001 - 100 1/s with decreasing measuring-point duration of 30 s – 2 s for OBM A, B, and C at 28 °C and 50 °C, measured with Anton Paar MCR 302.
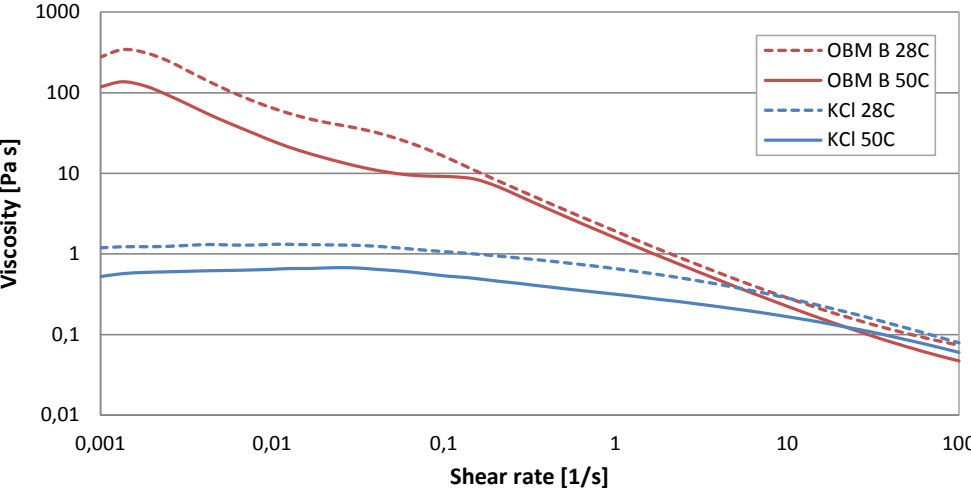
Figure 16. Low shear-rate flow curves in a shear-rate range of 0.001 - 100 1/s with decreasing measuring-point duration of 30 s to 2 s for OBM B and the KCl fluid at 28 °C and 50 °C, measured with Anton Paar MCR 302.

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Another possible explanation could be the one from Ackerson (1990) made during light scattering studies of suspensions of hard colloidal spheres. He found that for very low shear rates the Brownian motion positions the water droplets in a more regular structure in the mean, which then appears as a crystalline structures. This explanation was extended by Saasen (2002) while researching barite sag in oil-based drilling fluids. He found the crystalline structure to stay intact, as long as the Brownian motion showed a dominant behavior. When the shear rate increases, the Brownian motion is no longer able to reconstruct the crystalline structure of the water droplets and the individual droplets will then have to get redirected. This results in chaotic motion and an increase in viscosity which leads to the peak at very low shear rates. To get a more complete understanding of the case an extended investigation is needed. The measuring direction could be changed to conduct tests from high to low shear rates. Foss and Brady (2000) describe a competition between hydrodynamic forces and Brownian forces, where at very low shear rates, even lower than tested here, shear thinning can be observed.

● Water droplet ▢ Base oil
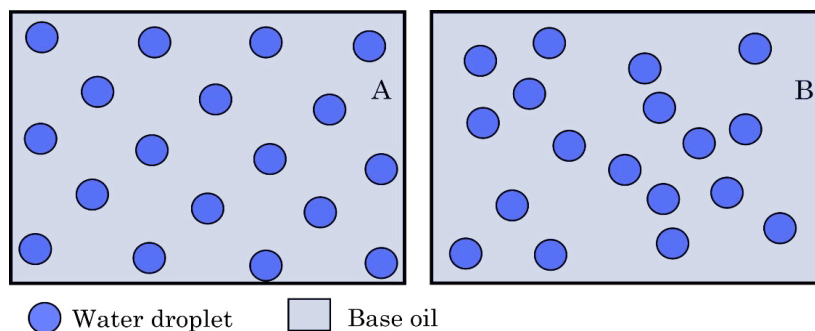
Figure 17. A) Self-arranged water droplets creating a crystal like structure at very low shear rates B) Chaotic motion results in redirected water droplets at higher shear rates.

2.3.3 Amplitude Sweeps

Figure 18 presents a comparison of the storage and loss moduli from amplitude sweeps for the OBMs A, B, and C at 28 °C and 50 °C. OBM C showed the highest G' and G" values, as well as the highest strain tolerance and the longest LVER for both temperatures. At 28 °C the G" of OBM A develops a peak just before the flow point, indicating an increasing portion of deformation energy. According to Mezger (2014), this deformation energy is initiating a breakdown of the internal structure already before the

final breakdown occurs. At 50 °C similar peaks can be seen also in the G' curves of OBM A and B. The peaks are occurring after the flow point and indicate an extra network structure being built.
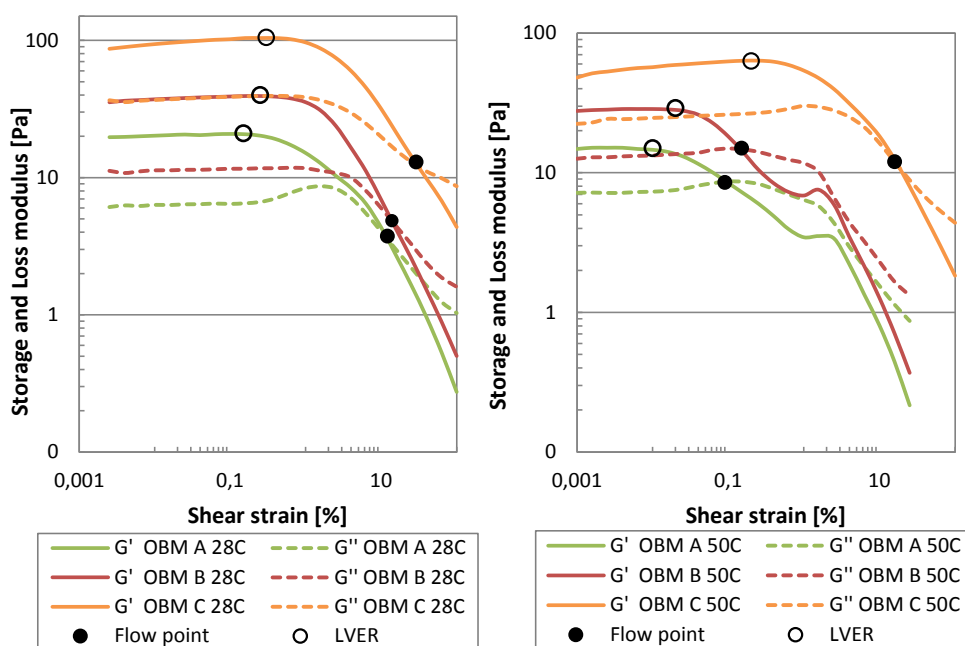
Figure 18. Storage and loss modulus of fluids OBM A, B, and C for 28 °C and 50 °C, measured with Anton Paar MCR 302. Flow point (filled circles) and the end of LVER (open circles) are marked.

Figure 19 shows a comparison of amplitude sweeps of the KCl fluid and the OBM B for 28 °C and 50 °C. The KCl fluid curves differ from the OBM curves. For both temperatures the storage modulus is lower than the loss modulus over the whole shear-strain range, and no cross-over point is found. Such characteristics indicate a dominant viscous behavior over the elastic behavior. Although no cross-over point exists, a flow point can still be found where the G' deflects from linearity. This behavior is typical for viscoelastic fluids such as polymer solutions. Their molecular chains are entangled (Figure 20) inducing some degree of structure in the fluid, but no consistent network of forces throughout the bulk. Table 7 contains G' and G" values at the end of the LVER, tan δ, and the LVER itself. Tan δ represents the loss factor, defined as the ratio of G" to G'. When tan δ>1, the sample shows a more viscous behavior, and respectively a more elastic behavior when tan δ<1. This can also be seen in Figure 21, where tan δ is plotted

over the shear strain range. The internal friction changes when the curve deflects from linearity.

The G' values of the OBM B have a constant value until a strain of about 10 % is reached. This reflects that the fluid is constructed as a pure dispersion and emulsion. As long as the strain is less than around unity, the particle and droplet positions are kept in a position trying to reach an energy minimum. When the strain becomes larger the particles and droplets start to leap frog; totally destroying the structure and hence decreasing the G' values. Here, the elasticity works on short range distances. The shear strain of the KCl fluid shows ca. 50 to 100 times higher values than the OBM B before the G' starts to deflect, indicating that the elasticity is not destroyed by rearrangement of the particle positions in the sample.
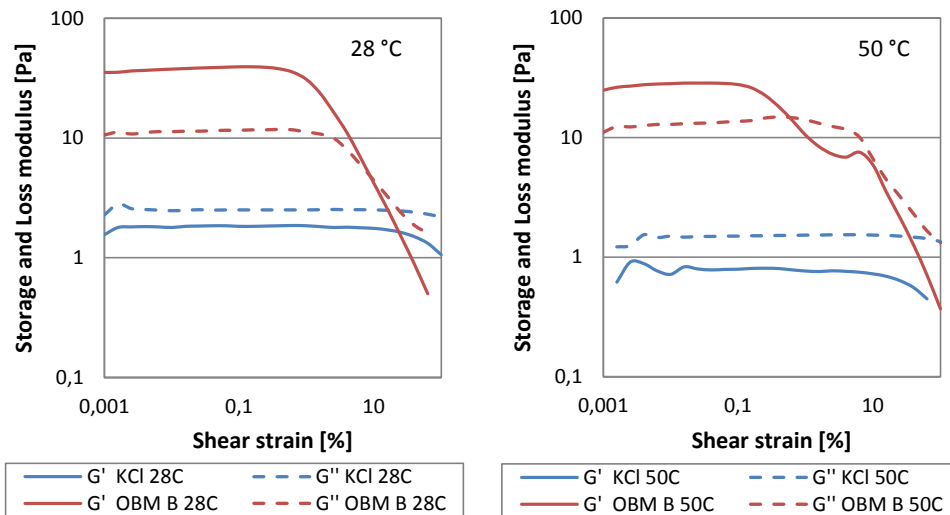
Figure 19. Amplitude sweeps showing the storage and loss moduli of the KCl fluid and the OBM B fluid for temperatures of 28 °C, and 50 °C, measured with Anton Paar MCR 302.

The domination of the loss moduli over the storage moduli, and the corresponding loss factor of tan δ >1 in the amplitude-sweep tests for the KCl fluid indicate a viscous character. This is in accordance with other results presented here. The OBM shows a dominant elastic behavior, indicating a microstructure in the fluid.
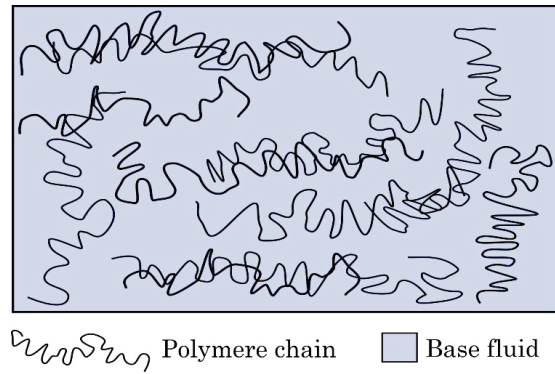
Figure 20. Illustration of entangled polymer chains in base fluid.
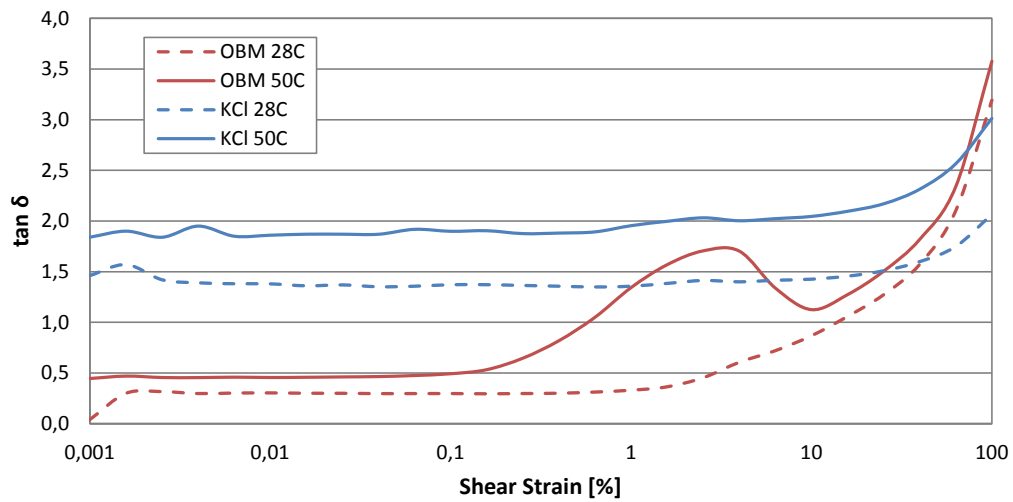
Figure 21. Ratio of the loss to the storage moduli plotted over the shear strain range.

Table 7. Storage and loss moduli, loss factor, and LVER of the OBMs and the KCl fluid for 28 °C and 50 °C.

Fluid	Temperature [°C]	G' [Pa]	G" [Pa]	tan δ	LVER (%/Pa)
OBM A	28	20.2	6.3	0.3	0.01/20.2
	50	14.1	7.4	0.5	0.06/14.1
OBM B	28	37.6	11.3	0.3	0.02/37.6
	50	27.8	13.7	0.5	0.1/27.8
OBM C	28	104	39.1	0.4	0.3/105
	50	63.4	26.4	0.4	0.2/63
KCl	28	1.8	2.5	1.4	6.3/1.8
	50	0.8	1.5	1.9	6.3/0.8

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2.3.4 Temperature Sweeps

Figure 22 presents temperature-sweep curves of all four fluids in a temperature range from 5 °C to 50 °C. The KCl fluid, the OBM A and the OBM B show an almost linear decrease in viscosity with increasing temperature, whereas OBM C decreases clearly nonlinearly. OBM C also shows the highest temperature dependency of the viscosity, probably due to a higher concentration of base oil in the fluid. A gas chromatogram of the base oil was made and revealed a high concentration of long chained hydrocarbons (C16 or higher). When lower temperatures are reached, these long chained hydrocarbons may start to crystalize and therefore increase the viscosity.
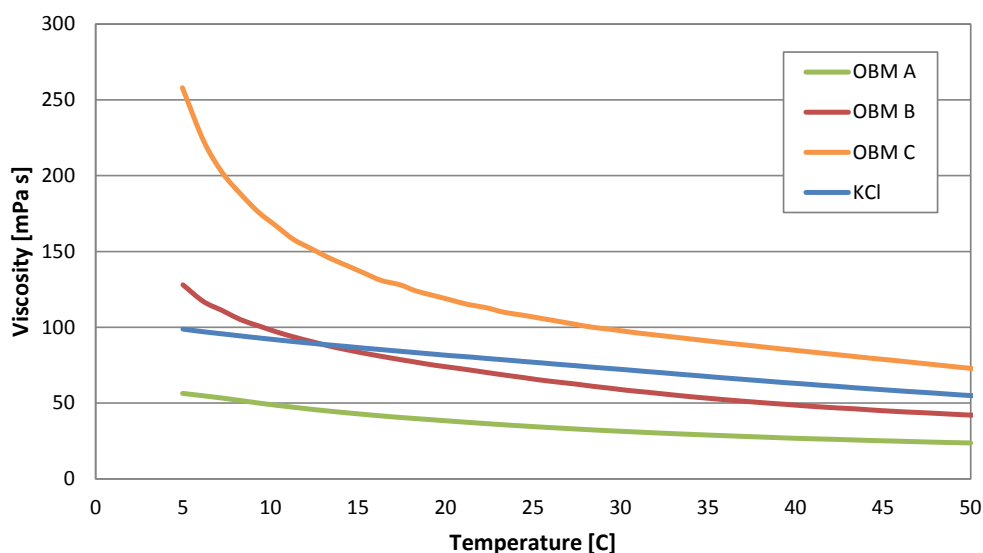
Figure 22. Temperature-sweep curves for OBMs A, B, C, and the KCl fluid measured with Anton Paar MCR 302.

2.3.5 Thixotropy

Figure 23 presents the storage and loss moduli for OBM A, measured with 3-interval-thixotropy tests. The rest interval was performed inside the LVER. During the recovery interval the fluid showed a structure buildup which exceeded the values from the rest interval. This happened for both temperatures. At 50 °C the storage modulus started to decrease after about 3000 s, indicating an unstable structure for the timeframe of this

test. After 7000 s the value of G' has dropped to 55.8 % of the maximum value. Only a slight decrease in storage modulus was observed for the 28 °C measurement, where G' was reduced to 91.7 % of the maximum value. Knowledge about structure development is important during operation when the fluid circulation has to stop. When structural decomposition happens too early, cuttings may settle and accumulate on the down side of the wellbore.

The slight decrease of the G' values for both temperatures during the initial rest interval are not quite understood. It could be that the changes in the fluid structure happen relatively fast. The structure rebuild after the first 200 s of the recovery interval is already at 100 % of the initial rest interval.
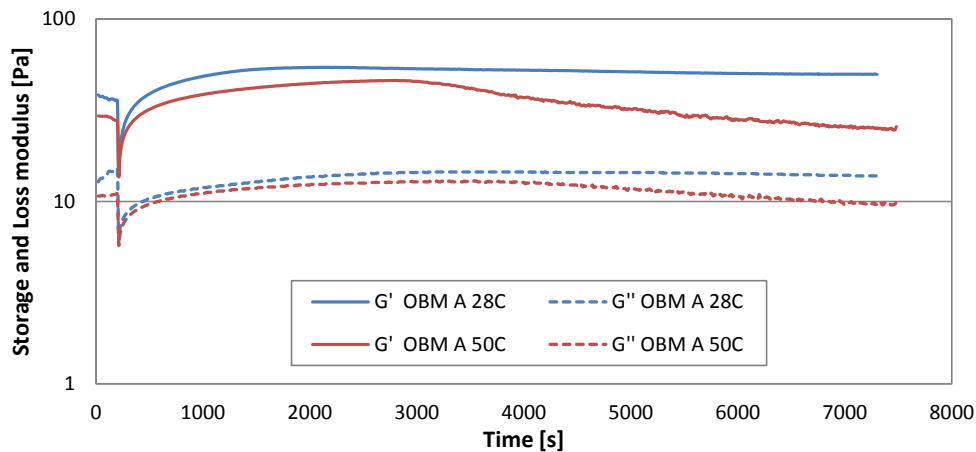
Figure 23. 3-interval thixotropy tests for OBM A at 28 °C and 50 °C measured with Anton Paar MCR 302.

In Figure 24 the results are shown for the KCl fluid. Similar to the observations from the amplitude sweeps, the storage modulus is also lower than the corresponding loss modulus. Interestingly, the storage modulus is increasing throughout the recovery interval and approaching the loss modulus. This happened for both temperatures. At 10 °C the curves are actually crossing each other. The cross-over point could not be determined directly because the G' and G" are crossing each other several times between 6000 s and 8000 s before the G' finally takes over. The test at 28 °C seems to have a more moderate structure increase. The temperatures of 10 °C and 28 °C were chosen because they represent each side of the crossing-point observed in the temperature-sweep test (Figure 22).
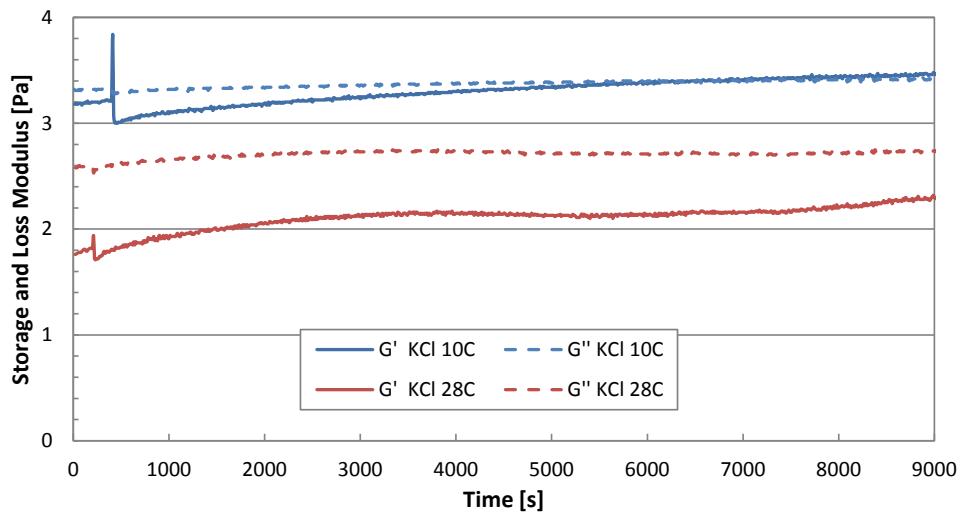
Figure 24. 3-interval-thixotropy tests for the KCl fluid at 10 °C and 28 °C measured with Anton Paar MCR 302.

2.3.6   Shear-Stress Sweeps

Figure 25 contains shear-stress sweeps of the OBMs at temperatures of 28 °C and 50 °C. All graphs show clear yield stresses where the curves deflect from linearity. The yield stresses are marked with dots. For 28 °C the yield stresses are higher than for 50 °C, and with increasing density the yield stress rises as well. The slight deflection before 1 Pa is probably due to a transition state and can be disregarded.

The controlled shear-stress sweeps for the KCl fluid (Figure 26) develop a linear plateau and do not deflect from that in the higher shear-stress area, therefore no yield stress can be determined. This is in conformity with the results from the amplitude-sweep tests, where the crossing point (flow point) of the storage and loss moduli can be interpreted as a yield stress. During the current measurements no crossing point was found due to the dominating loss modulus, hence no yield stress could be determined either. The decrease of viscosity and elasticity with increased temperature was also observed by Bui et al. (2012).
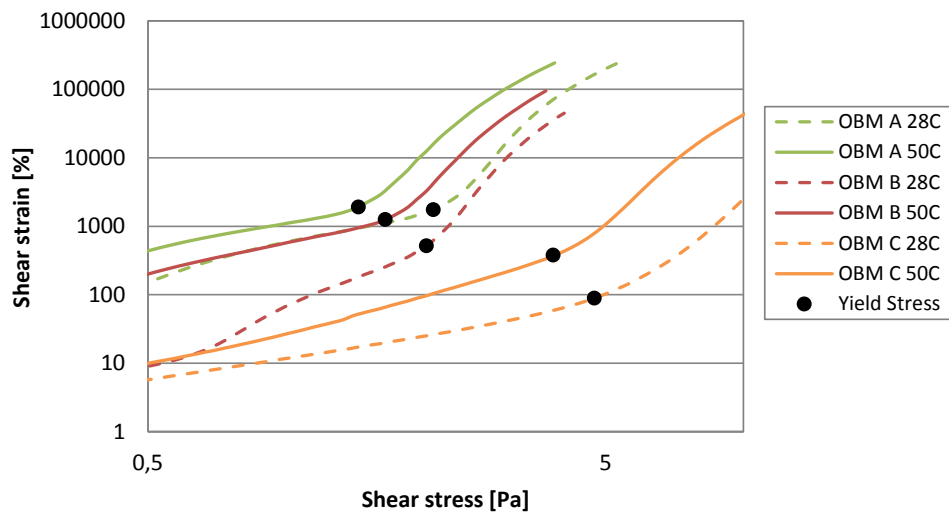
Figure 25. Shear-stress sweeps of OBM A, B, and C for temperatures of 28 °C and 50 °C, measured with Anton Paar MCR 302.
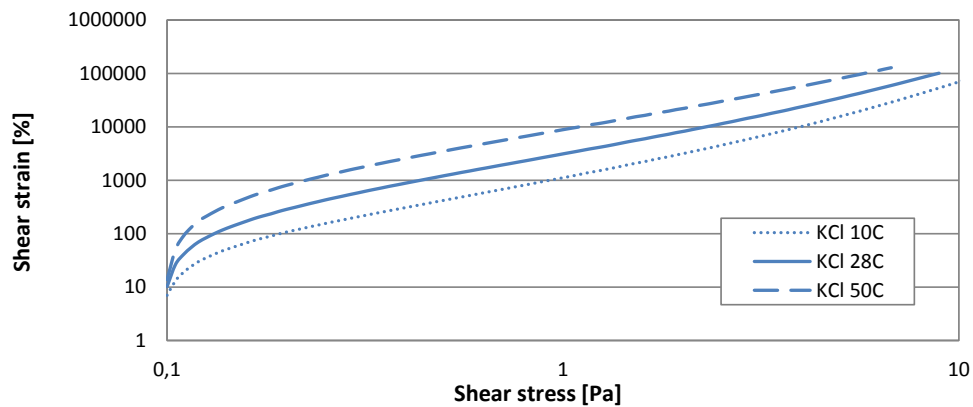
Figure 26. Shear-stress sweeps of the KCl fluid for temperatures of 10 °C, 28 °C, and 50 °C, measured with Anton Paar MCR 302.

2.3.7 Yield Stress

The yield stress is an important parameter characterizing drilling fluids in static condition. The value of the yield stress is not a material constant and strongly dependent on the measuring method and/or the regression method. Here three different common methods were used to compare results. Method 1 (M1) was a shear-stress sweep. The

yield stress can be found in the resulting shear strain vs shear stress plot, where the curve deflects from linearity. Regression with the Herschel-Bulkley model was used as method 2 (M2). The third method (M3) were amplitude-sweep tests, where the crossover points of the storage and loss moduli were used. In amplitude-sweep tests two possibilities exist for determining the yield stress. The end of the LVER is the beginning of the structural degradation, but still a rest network structure is present. First after the crossover point (G'=G") the fluid is flowing as a whole and the viscous forces are dominating. Using the crossover point is a more practical approach, as from this point on the structure is broken to an extent that the fluid actually flows. The range between the end of the LVER and the crossover point is also called the yield zone (Mezger 2014).

Table 8 shows the yield-stress values for the OBMs at 28 °C and 50 °C. The KCl fluid did not show a yield stress. For the higher temperatures the values are consistently lower for all three fluids. The highest values were obtained with method 2. For OBM A and B the difference between the methods was the least between M1 and M2, while M3 showed much lower values. For OBM C the M3 values did not deviate to such an extent.

Table 8. Yield stresses of OBM A, B, and C for temperatures of 28 °C and 50 °C determined by three different methods, M1 – shear-stress sweep, M2 – regression with Herschel-Bulkley model, M3 – amplitude-sweep test.

Fluid	OBM A		OBM B		OBM C	
Temperature [°C]	28	50	28	50	28	50
Yield stress M1 [Pa]	2.1	1.4	2.0	1.7	4.7	3.8
Yield stress M2 [Pa]	2.9	1.8	3.3	2.2	9.9	4.8
Yield stress M3 [Pa]	0.61	0.05	0.95	0.12	5.4	2.9

2.4 Results of the Flow-Loop Experiments

Figure 27 shows the sand holdup for different average fluid velocities for experiments with and without drill-string rotation. A high impact on cuttings removal can be seen for experiments with a drill-string rotation of 150 rpm for both fluids. The drill-string rotation is dominating the flow related properties. The rotation is destroying the particle interactions and therefore reducing the resistance of the bed. Additionally the turbulences created by the rotation counteract the resettling of the sand particles in the test section.

At a fluid velocity of 1.2 m/s and without drill-string rotation, the difference in sand holdup is largest between OBM B and the KCl fluid, and OBM B gave a 5% lower sand holdup than the KCl fluid. OBM B removes the sand bed better, leaving less sand in the test section for all tested fluid velocities, without drill-string rotation.
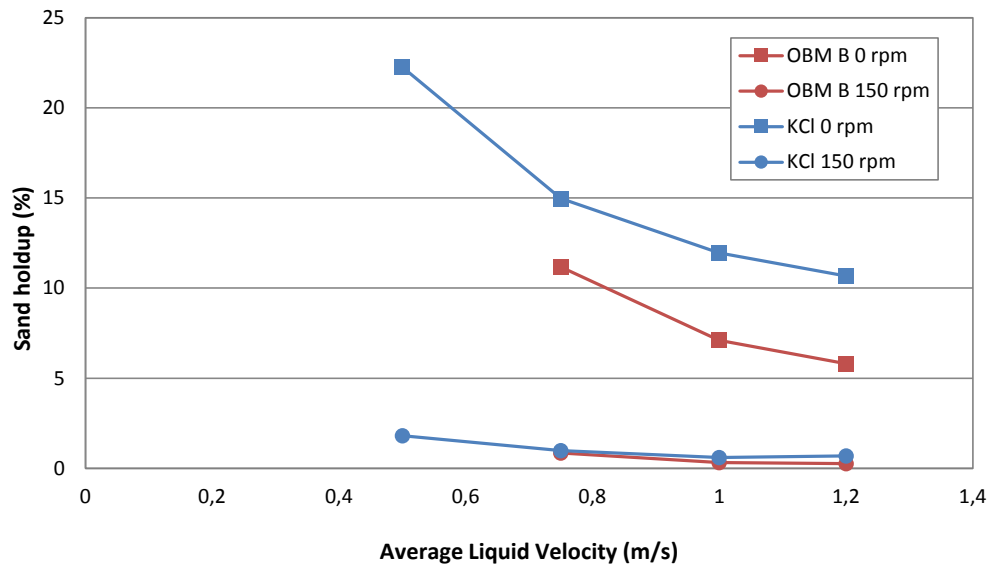

Figure 27. Sand holdup versus superficial liquid velocity for the KCl fluid and OBM B with and without drill-string rotation (OBM B data collected from Sayindla et al. 2016, KCl fluid data from Sayindla et al. (Submitted))

When evaluating the results seen in the flow-loop experiments (Figure 27), two cases can be differentiated. One case with drill-string rotation and higher flow velocities, and one case without drill-string rotation and lower flow velocities. Considering the lower flow velocities, the importance of fluid interaction with the cuttings while flowing becomes less relevant, and the cutting-beds resistance to erosion becomes more important. The viscosity profiles of the KCl fluid and the OBM B are similar in the relevant shear-rate range of the flow-loop experiments, but different rheological behavior could be seen in the rheological investigation. The interpretation of the results of the low shear-rate rheology experiments helps to understand the suspension characteristics and the differing cuttings-transport behavior of the fluids.

In experiments without drill-string rotation OBM B showed a better performance. This can be explained by how the fluids affect the sand bed and influence its resistance to erosion. The OBM B is a water in oil emulsion with a yield stress, and the KCl fluid is

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built as a polymer in brine suspension (Table 3). The polymers and the water in the KCl fluid increase the interactive forces in the sand bed by creating stronger inter-particle forces, which further increase the resistance to erosion. During amplitude sweeps (Figure 19), the KCl fluid showed that it can tolerate up to 100 times more strain deformation than the OBM B. This elasticity holds back the sand particles and makes entrainment in the fluid flow more difficult. Saasen (1998) found similar behavior in fluids containing xanthan gum. In case of the OBM B, the yield stress is responsible for the strength of the sand bed. This yield stress is comparably weaker than the elasticity in the KCl fluid. Cuttings erosion from the sand bed with OBM B is thus easier, and the sand particles are released more immediately. A schematic drawing is presented in Figure 28 where the thick black arrow represents the yield stress for the case of OBM, and the thinner spring-like arrows represent the elasticity in the KCl fluid. Additionally the amplitude sweeps for each fluid and the flow curves are shown.
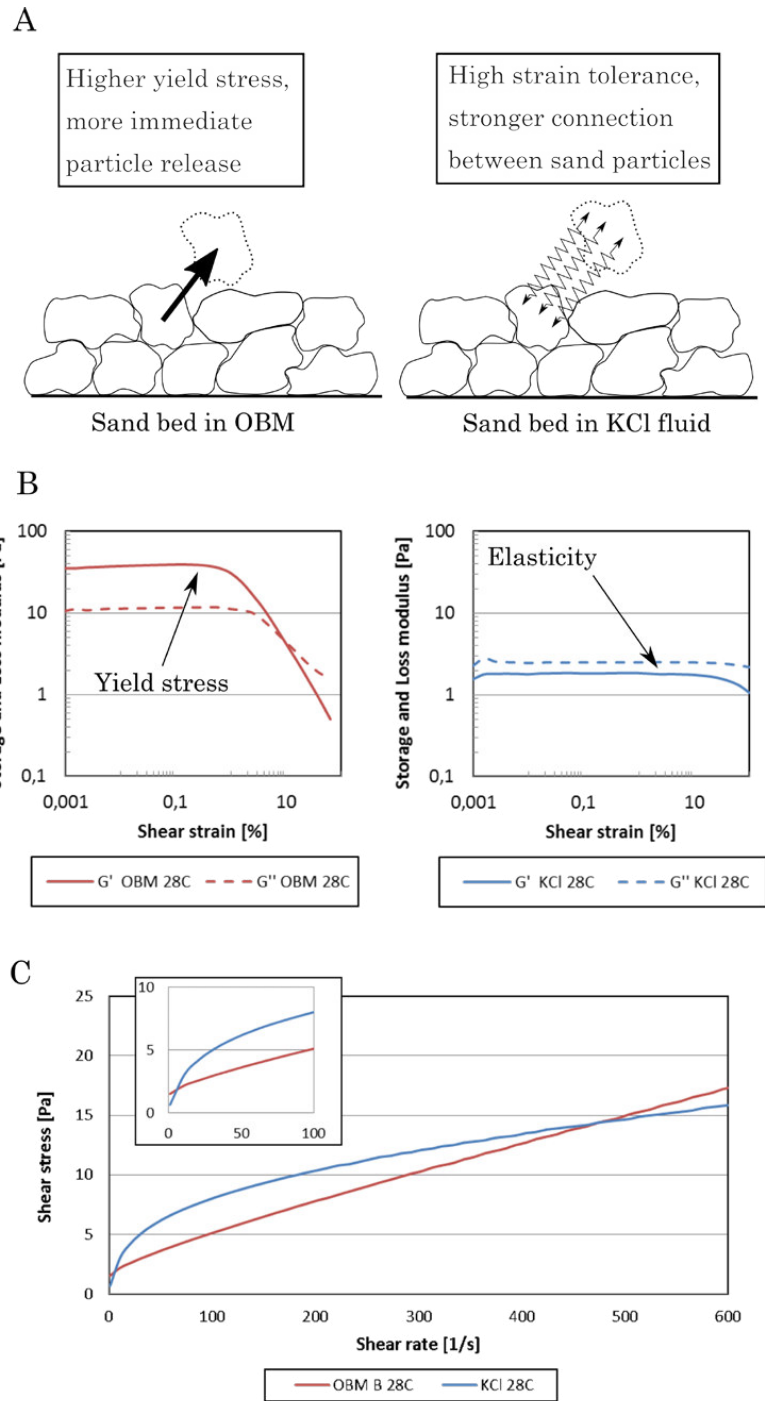
Figure 28. Schematic drawing presenting A) the cuttings behavior in the OBM B and KCl fluids when exposed to low flow conditions together with B) the amplitude sweeps showing the yield stress in the OBM and the elasticity in the KCl fluid, and C) the flow curves

3. Conclusion

Parameters influencing hole cleaning are manifold. The prediction of hole cleaning cannot be done from a single flow or rheological parameter alone and linking all the parameters together also requires to understand their individual behavior. The rheological analysis of this thesis involved characterization of oil-based and water-based drilling fluids with Anton Paar rheometers measuring different flow curves, amplitude sweeps, temperature sweeps, and thixotropy. A Fann 35 viscometer was used to keep control of the fluid condition during periods of flow-loop experiments. During these flow-loop experiments the hole-cleaning capabilities of drilling fluids were investigated and the oil-based drilling fluid showed superior cuttings-transport abilities.

The OBMs of this study are water in oil emulsions with a yield stress and a linear viscoelastic range indicating a light structure in the low shear-rate range. The KCl fluid is a polymer-containing water-based fluid with a strain tolerance up to 100 times larger than the OBM.

The results of the comparative flow-loop experiments suggest that emulsion based fluids, exhibiting yield stresses and light internal structures at very low shear rates, are the better option for hole cleaning. In a cuttings bed created in OBM B, the yield stress of the fluid may initially require higher forces to remove a cutting from of the bed, but when the yield stress is overcome the cutting will immediately enter the fluid flow. On the other hand, the larger elasticity and strain tolerance in the KCl fluid, caused by the polymers, will hold back the cutting and thus making it more difficult to remove the cutting from the bed and entrain the fluid flow.

High-velocity fluid flow and drill-string rotation are certainly two dominating parameters for the removal of cuttings from a wellbore. The flow-loop experiments with drill-string rotation demonstrated the dominating effects of the drill-string rotation over the flow-related properties during cuttings transport. The particle interactions were destroyed and the resistance of the sand bed reduced. With a lower resistance the particles were easier brought into the fluid flow and cuttings transport improved. Turbulences created by the drill-string rotation and fluid flow may in addition counteract the particle settling.

The main concluding points are:

- The viscoelastic properties of the tested drilling fluids seem to have only little impact on the cuttings transport. They are more likely to have an impact on the strength of the cuttings bed and its resistance to the drilling-fluid flow.
- The results of the comparative flow-loop experiments suggest that emulsion-based fluids, exhibiting low yield stresses and light internal structures at low shear rates, are the better option for hole cleaning.
- The resistance in a cuttings bed is affected by the yield stress of the drilling fluid.
- The resistance in a cuttings bed is affected by the elasticity and strain tolerance in the drilling fluid. This effect is stronger than the yield-stress influence.
- The performance of OBM B may profit from the absence of polymers that otherwise consolidate the cuttings bed and make the cuttings removal more difficult.
- Drill-string rotation will dominate the flow-related properties.

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Appendix

A.1 Additional Results of the Preconditioning of Oil-Based Drilling Fluids

Figure 29 shows dial readings of OBM C at a temperature of 50 °C without pre-shear. The dial readings are slightly increasing with increasing waiting times, especially for rotational speeds of 600 to 100 rpm. The 6 and 3 rpm measurements appear rather stable as well as the 10 sec and 10 min gel strength measurements. This may be explained by the fact that at the time of these last readings, the fluid has already been sheared significantly (through the 600 to 100 rpm measurements). Figure 30 shows dial readings of OBM C at a temperature of 50 °C with pre-shear. A much flatter trend with increasing time from sampling is apparent with pre-shearing than without pre-shearing. Maximum structure build-up values are 4 % (600 rpm) for 28 °C and 5 % (200 rpm) for 50 °C. In Figure 30 the measurements after 2 hours deviate clearly from the rest. There is no apparent explanation for this and these measurements are considered less reliable. Note that readings after 24 hours show a higher value than the starting value, indicating that after 24 hours waiting time pre-shearing does not reproduce the initial state. The 10 s and 10 min gel strength measurements are nearly unaffected by the pre-shearing.
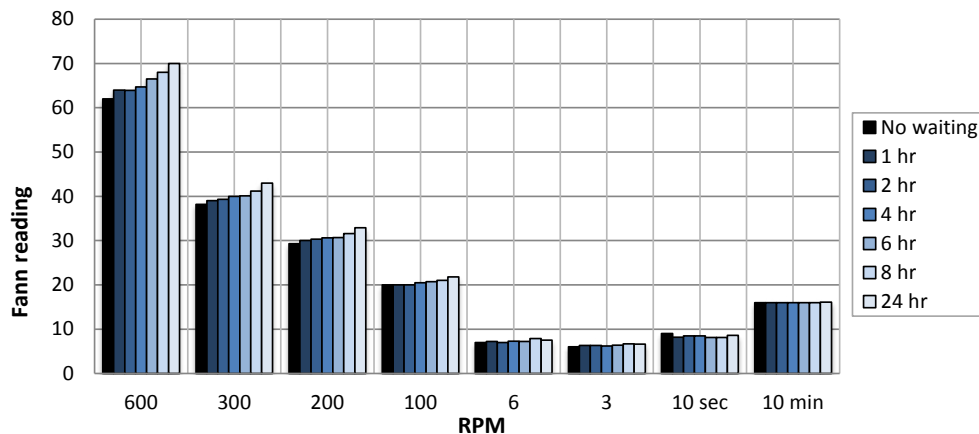
Figure 29. Fann 35 dial readings of OBM C taken without pre-shear at a temperature of 50 °C. Waiting times as indicated, the lighter the color, the longer the waiting time.
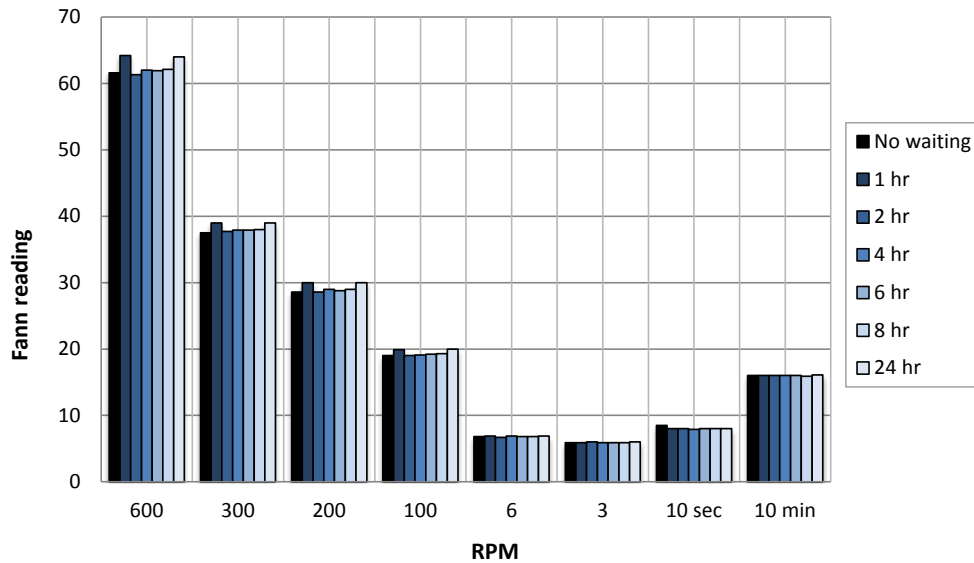
Figure 30. Fann 35 dial readings of OBM C taken with pre-shear at a temperature of 50 °C. Waiting times as indicated, the lighter the color, the longer the waiting time.

Figure 31 and Figure 32 show flow curves measured in the Anton Paar rheometer, at 28 and 50 °C, respectively. The top bunch of lines in the figures represent flow curves with no pre-shearing. In Fig. 6, the structure build-up with no preshear at 28 °C is clearly visible with increasing waiting times. The lower bunch of lines, representing measurements with preshear fall almost on top of each other, i.e. the same trend is apparent here as for the Fann measurements. The measurement for 8 hour rest and preshear clearly deviates from the rest and is considered an outlier.

Figure 32 shows the same measurements done at 50 °C. At this temperature the structure regeneration with no preshear is much less pronounced, and the difference between the pre-sheared and not pre-sheared measurements is small. In other words, at 50 °C pre-shearing has no significant influence on the flow curves.
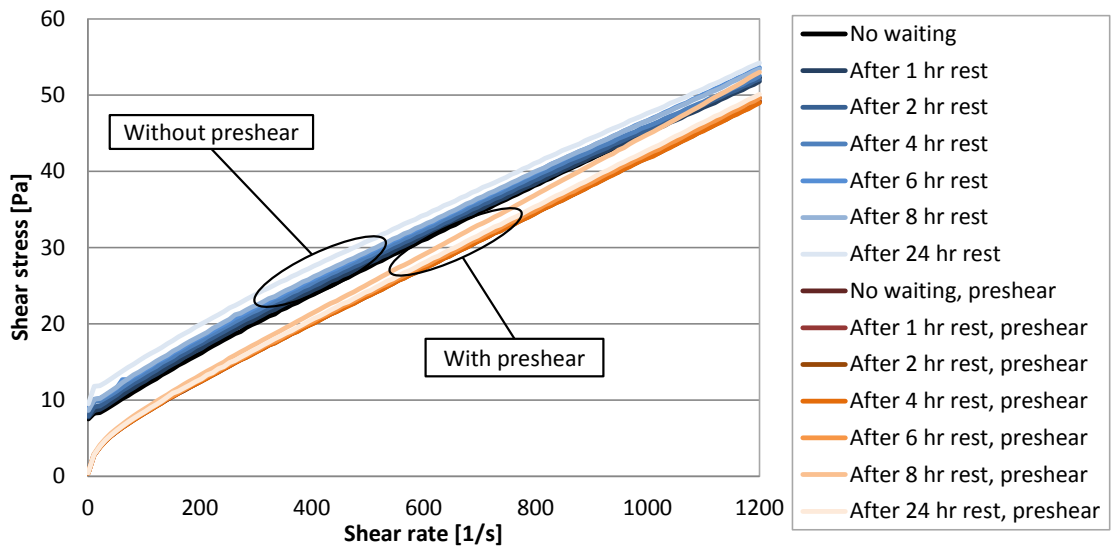
Figure 31. Flow curves of OBM C at 28 °C. Waiting times as indicated, the lighter the color, the longer the waiting time, measured with Anton Paar MCR 302.
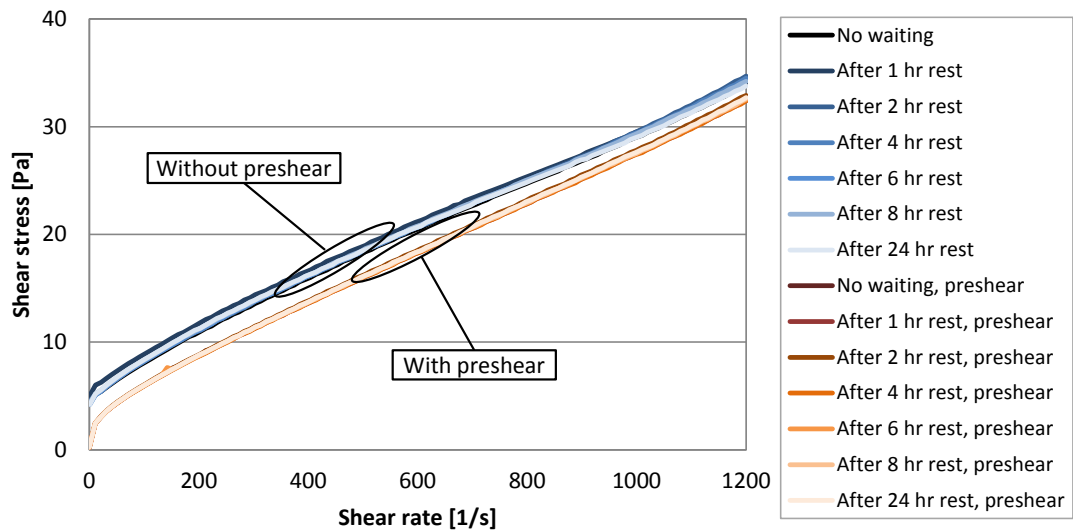
Figure 32. Flow curves of OBM C at 50 °C. Waiting times as indicated, the lighter the color, the longer the waiting time, measured with Anton Paar MCR 302.

B.1 Additional Results of the Preconditioning of Model Drilling Fluids

Generally, the fluids show shear thinning trends, and experiments with preshear and without waiting time led to the lowest viscosities. The biggest difference in viscosity can be seen between the measurements without waiting time and a waiting time of 24 h, especially for fluids #1a and #2a. Small amounts of salt increase the viscosity, this applies both with and without xanthan gum present in the composition, see Figure 33 - Figure 36.

A high salt concentration of 12 g/l NaCl reduced the viscosity of sample #2c, compared to #2b (Figure 36).

Figure 33 - Figure 36 show that the pre-shearing procedure is not sufficient to obtain results independent of waiting time for fluids #1 and #2. However, pre- shearing is much more efficient when 0.6 g/l NaCl is added, as seen in Figure 34 and Figure 36. The measurements with fluid #1a and #1b were also conducted at 50 °C and show the same qualitative trends as at 24 °C.

Interestingly, the experiments with fluid #2, which include xanthan gum, show similar trends as fluids #1. For fluids without salt and measurements without preshear the viscosity is much lower.

As expected, preshear does not have much effect on the result without waiting time.
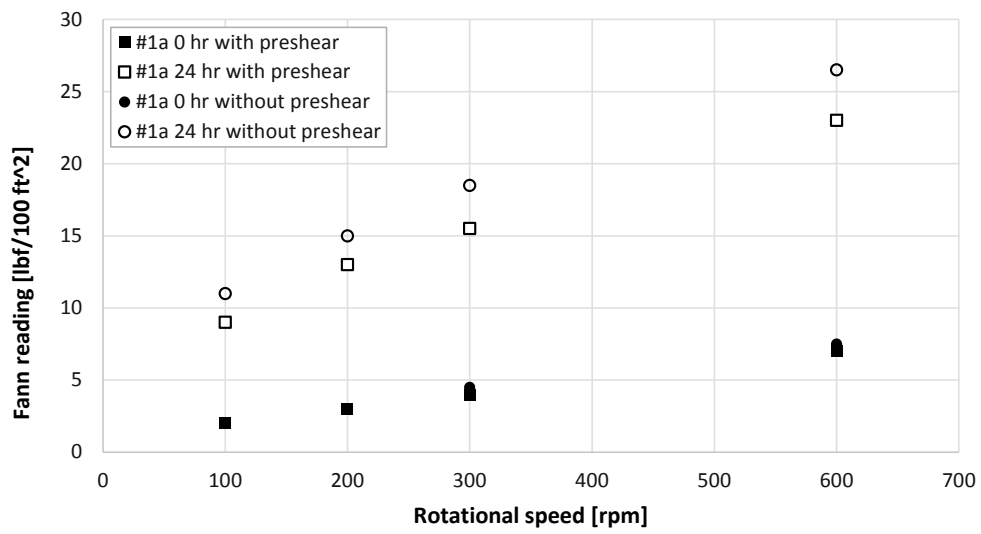
Figure 33. Fann 35 measurements for the laponite suspension #1a at 24 °C
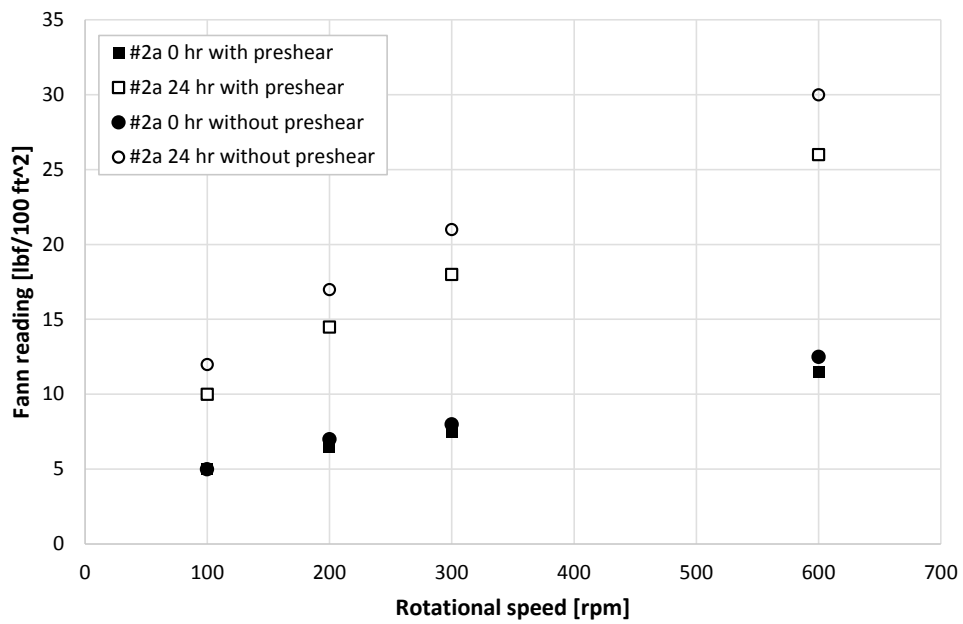
Figure 34. Fann 35 measurements for the laponite suspension #2a at 24 °C.

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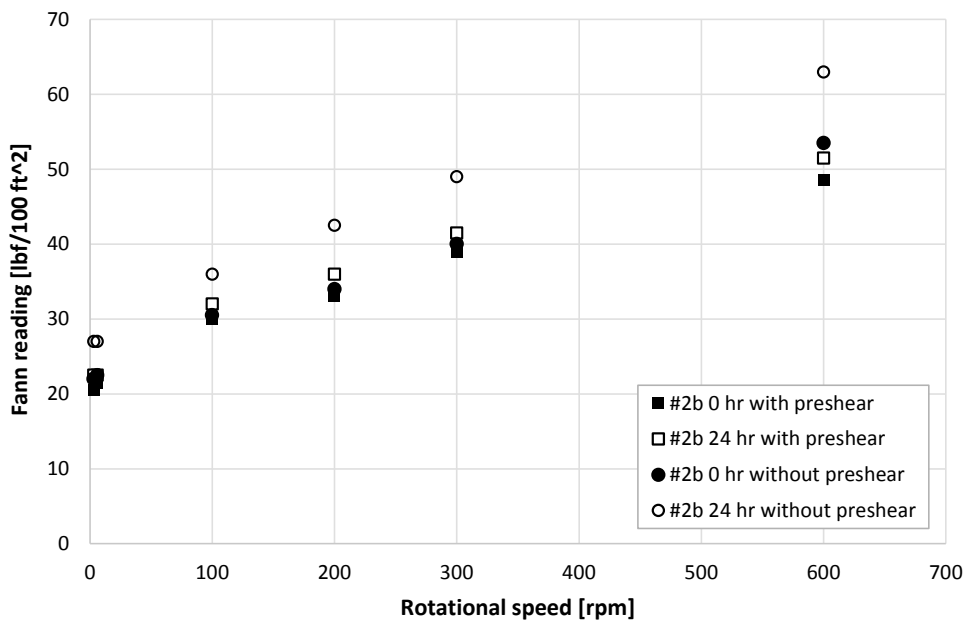
Figure 35. Fann 35 measurements for the laponite suspension #2b at 24 °C.
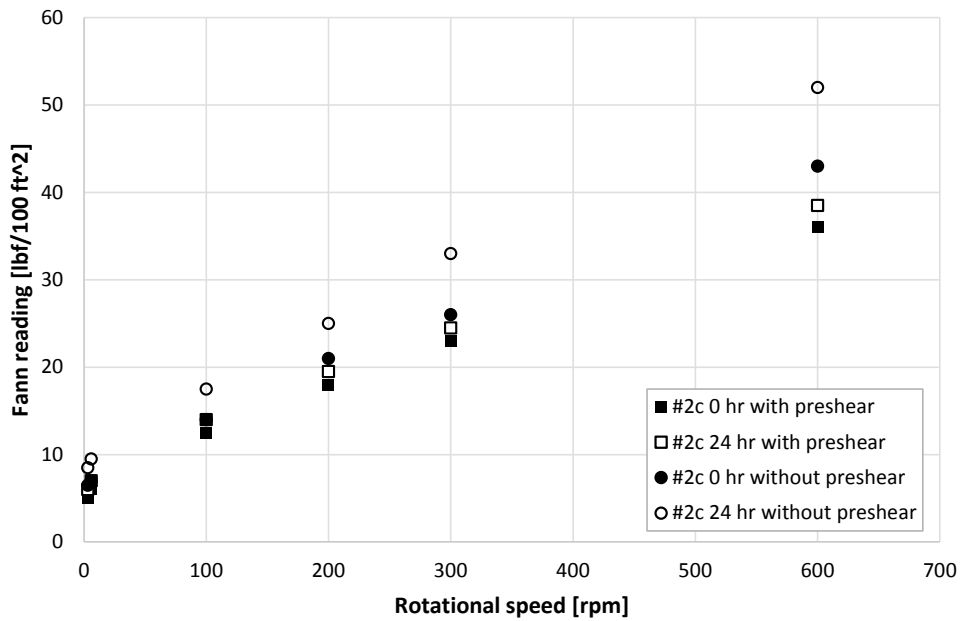
Figure 36. Fann 35 measurements for the laponite suspension #2c at 24 °C.

The observations made from the Fann measurements are confirmed by the flow curves generated from Anton Paar measurements (Figure 37 - Figure 41).

The flow curves measured with the Anton Paar rheometer show apparent shear thickening trends in the samples without preshear for shear rates >800 1/s (Figure 41). This trend is more pronounced for samples with a waiting time of 24 h and higher NaCl concentrations and especially predominant for fluid #2c, which contains xanthan gum and a high concentration of salt. This effect might be a result of the thixotropic behavior, because the measurements start at high shear rate and are affected by a shear history.
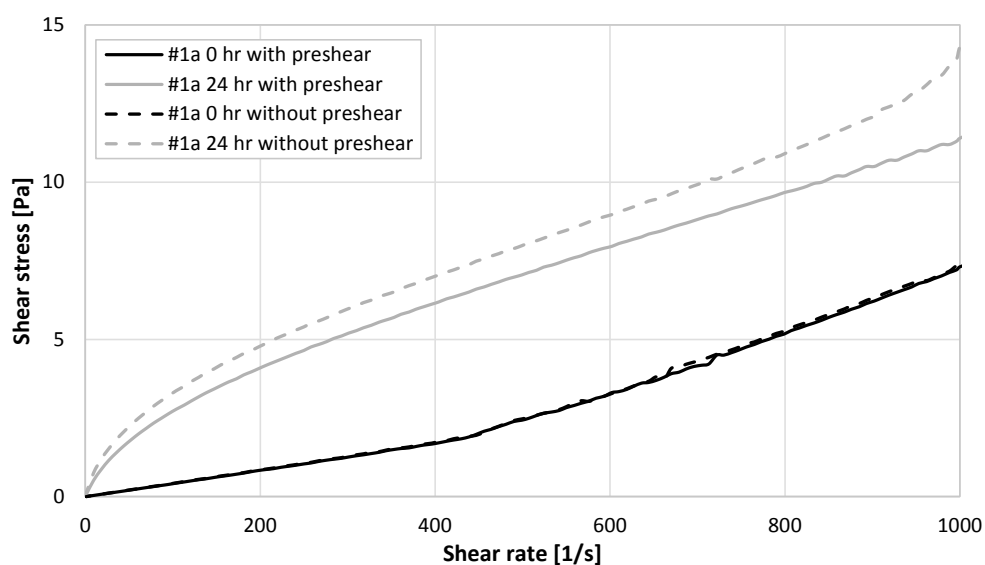
Figure 37. Anton Paar flow curve measurements for fluid #1a at 24 °C.
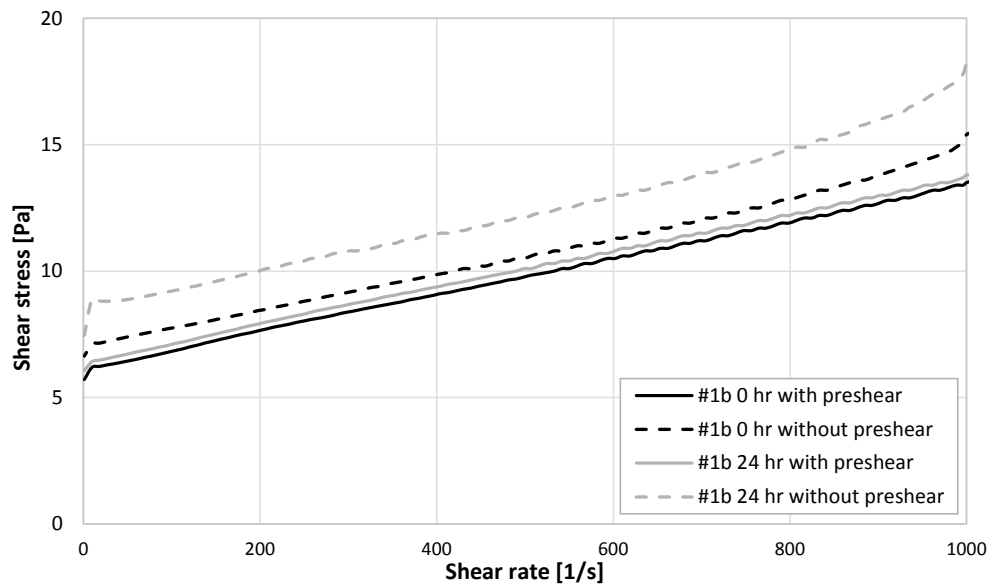
Figure 38. Anton Paar flow curve measurements for fluid #1b at 24 °C.
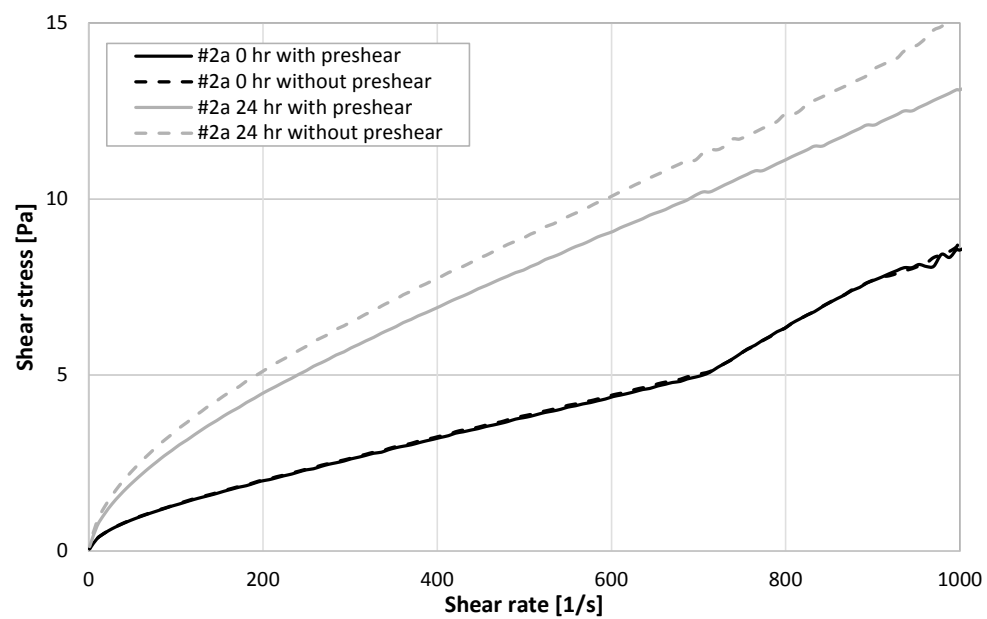
Figure 39. Anton Paar flow curve measurements of fluid #2a at 24 °C.
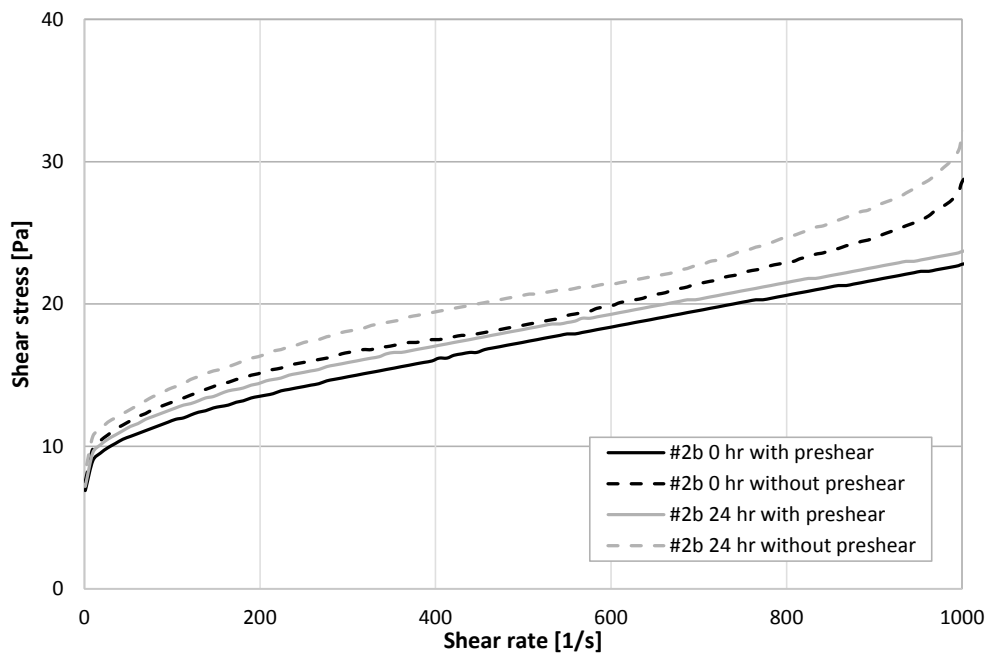
Figure 40. Anton Paar flow curve measurements of fluid #2b at 24 °C.
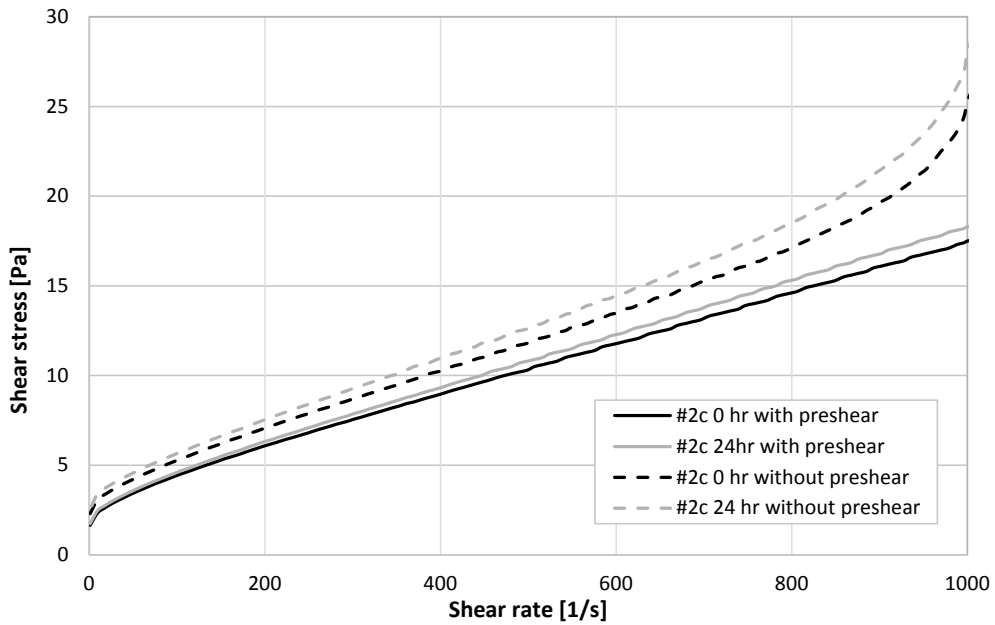
Figure 41. Anton Paar flow curve measurements of fluid #2c at 24 °C.

C.1 Articles

Paper 1

Establishing an Experimental Preconditioning Procedure for Rheological Characterization of Oil-Based Drilling Fluids

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ABSTRACT

In the oil industry the ISO 10416/ISO 10414-2 standards, which are used for determination of rheological properties of oil-based drilling fluids, do not in detail specify how the fluids should be pretreated before measurements. In this study, a systematic approach is used to quantify the influence of waiting time and/or pre-shearing on measurements of viscosity and other rheological properties of an oil-based drilling fluid.

INTRODUCTION

Oil-based drilling fluids (OBDFs) are thixotropic fluids, meaning that their properties may change with time. One also knows that the fluid properties of OBDFs are highly dependent on shear history. As a result of this, it is important to have a consistent procedure for how to treat the fluids prior to measurements. This is vital in order to be able to compare experimentally determined flow properties, not only in this project but also to enable comparison of results between labs.

Fluids involved in oil-industry drilling operations range from sea water and drilling fluids to well cements. Well cements are chemically reactive, with a pump ability time that has to be adjusted to the practical pumping operation. Therefore, in order to evaluate viscous properties of the well cement slurries strict preconditioning procedures exist to simulate the shear history of a cement prior to entering the annulus. These procedures include how to mix the cements slurry followed by a detailed procedure on how to measure the viscosity. These procedures can be found in publications by Guillot[1] and by Dargaud and Boukhelifa[2] or in API Recommended Practices[3].

For the drilling fluid industry, a similar degree of detailed procedures does not exist for determination of the fluid viscosity values. The ISO 10414-2/ API 13B-2[4] and ISO 10416/API 13I[5] standards are used for determination of viscosity and gel strength of drilling fluids by use of direct-indicating viscometers (Fann 35 viscometers). However, the drilling fluid standards do not in detail specify how the fluids should be pretreated before measurements. Often the pretreatment only consists of simply shearing the sample for a specific time at $1022 \ s^{-1}$, as performed by for example Maxey et al.[6] who sheared the sample for

two minutes at their measurement temperature. Further, if one wants to compare results from Fann 35 viscometers to measurements done on a rheometer, it is even more important to have a consistent pretreatment of the fluids. The questions one seeks to answer in the present study are: i) how large is the effect of preshearing/no preshearing/rest?; ii) is preshearing or rest the most preferable in order to get reproducible results? These questions are likely to become even more important if other rheological properties than viscosity are evaluated.

Bui et al.[7] presented a study concerning rheological properties of oil-based drilling fluids. The preparation procedures are not thoroughly described in the article. However, in another work Bui[8] presented this preparation in some more detail. In summary his procedure was to blend the drilling fluid portion, shear it at 1000 s^{-1} for 10 minutes and then let it rest statically for a definite time period. This time period was determined by measuring the linear viscoelastic properties to determine the time to reach an accepted level of stationary values. This time was then used in the other experiments.

Understanding the effect on rheological properties of drilling fluids based on activities performed before the measurements are taken is important also in the field. In practice fluid data are taken during different activities such as: drilling and circulation (high shear), tripping in/out (low shear), reserve volume preparations (low to no shear), etc. These data are often put in the same context and one searches for changes to the fluid based on trend analyses. Also knowing that the activity level on a drilling rig is high, the time from sampling until the measurements are done in the laboratory is varying and rarely documented. In this work, effects that may increase the variance of the data and also lead to wrong interpretation of data and trends are identified.

In the following, a methodical study is presented in which the effects of waiting time and/or preshearing on measurements of viscosity and other rheological properties of an oil-based drilling fluid are quantified in a systematic way. These results give a foundation for a suggestion for an experimental preconditioning procedure for rheological characterization of oil-based drilling fluids.

EXPERIMENTAL

Drilling fluid design

The fluid selection was based on previous work[9] and delivered by M-I SWACO. The oil-based drilling fluid (OBDF) was a field fluid which had been used during actual drilling operations. Prior to delivery, the fluid was cleaned, reconditioned and shipped to the research facilities of SINTEF. The OBDF is an emulsion of high-alkaline brine droplets in the continuous phase of base oil, and enriched with barite weight material as well as clay (Bentone128), emulsifier and fluid loss material. Bentone128 was used as the primary viscosifier. The original ratio of base oil to water, before clay addition, was 85/15. The fluid was used for circulation in a full scale flow loop, and while running through the sand removal filters of the circulation unit both sand, clay and small amounts of water were filtered out on each circulation. This, together with evaporation effects, lead to loss of water and a change in the oil/water ratio (OWR) over time. This dewatering effect was noticed over a few days of operation, but it was decided to continue with the operation and keep the viscosity expressed with Fann 35 measurements more or less constant. Bentone128 was added to compensate for the loss in viscosity, and at the respective time of sampling for the data measurements in this work, the OWR of the fluid was 91/9 for the first batch and 95/5 for the last batch.

Fluid characterization

Three batches of the OBDF were sampled from the flow loop at different times. The first two batches were sampled on March 20th (OWR 91/9) and April 8th 2015 and used for experiments on the Fann 35 viscometer. The third batch was sampled on April 20th 2015 (OWR 95/5) and used for measurements in the Anton Paar Physica MCR302. It should be emphasized that even though the three fluid batches might have slightly different OWR, the rheological properties of the batches are nearly identical.

Density measurements for the OBDFs were done by a standard Brand pycnometer. All three batches were measured to 1,26±0,01 g/ml.

OWR was measured by retort analysis, ref to ISO 10414-2/API 13B-2[4].

The effects of waiting time and/or pre-shearing were studied using the following time test matrix for measurements: immediately and after 1 hr, 2 hr, 4 hr, 6 hr, 8 hr and 24 hr resting. All measurements were performed both at 28 ⁰C and at 50 ⁰C, and the samples for 28 °C testing were stored at room temperature (approx. 20 °C), and the samples for 50 °C testing were stored in a heat cabinet at 38-42 °C. The whole matrix was repeated a second time with 10 min pre-shear preceding each measurement. The full test matrix was conducted both using a Fann 35 viscometer and an Anton Paar Physica MCR302 rheometer.

For the Fann 35 viscometer measurements were started immediately after sampling from the active flow loop. The measuring cup was heated to the required temperature by use of OFITE Thermocup 130-38-25. Temperature was at all times observed by use of Eurotherm 2408i Indicator unit, with precision down to 0,01 ⁰C. Viscosity and gel strength were measured following the ISO 10414-2/API 13B-2[4] and ISO 10416/2008[5] (600 -300-200-100-6-3 rpm, 10 sec and 10 min gel). For the preshearing measurement sequence, a shear rate of 600 rpm was applied for 10

min before starting the measuring sequence.

The MCR302 rheometer is equipped with an electrically heated temperature chamber. Before each test, the temperature was set with an accuracy of 0.01 ⁰C. To ensure temperature equilibrium an additional 7-8 minutes waiting time was added before start of measurements. A concentric cylinder measuring system (CC27) was chosen to avoid evaporation at 50 ⁰C, and the sample was changed for each new measurement. The fluid batches were mixed thoroughly every morning in a Hamilton Beach blender, at appr. 13000 rpm for 10 minutes. The measuring sequences following the previously described test matrix were then conducted immediately after mixing (for both 28 and 50 ⁰C). For each slot in the test matrix, flow curves and amplitude sweeps were performed. Flow curves (controlled shear rate) were recorded from shear rate 1 to 1200 s^{-1}. The amplitude sweep tests were conducted with a constant frequency of 10 s^{-1} and with increasing strain from 0.001 to 100 %. The whole test sequence was then repeated with a 10 min preshear at 1000 s^{-1} before each measurement. This shear rate corresponds to 600 rpm shear in the Fann viscometer.

RESULTS

Figures 1 and 3 show dial readings in the Fann 35 viscometer for 28 and 50 ⁰C, respectively, with no preshearing. For both temperatures, dial readings are slightly increasing with increasing waiting time, especially for rotational speeds of 600 to 100 rpm. A maximum structure build-up of 9 % for the 600 rpm reading at 28 ⁰C and 12 % at 50 ⁰C can be seen for the 24 hour time period. For the readings of 300, 200 and 100 rpm the structure build-up accounts to 2 – 10 %. The 6 and 3 rpm measurements appear rather stable as well as the 10 sec and 10 min gel strength measurements. This may be explained by the fact that at the time of these last readings, the fluid has already

been sheared significantly (through the 600 to 100 rpm measurements).

Fann 35 dial readings, for which the fluid has been pre-sheared for 10 min at 600 rpm (equivalent to 1022 s^{-1}) prior to measurements, are shown in Fig. 2 and 4 for 28 and 50 ^{o}C, respectively. A much flatter trend with increasing time from sampling is apparent *with* preshearing than *without* preshearing. Maximum structure build-up values are 4 % (600 rpm) for 28 ^{o}C and 5 % (200 rpm) for 50 ^{o}C, see Fig. 2 and 4. In Fig. 4 the measurements after 2 hours deviate clearly from the rest. There is no apparent explanation for this and these measurements are considered less reliable. Note that readings after 24 hours show a higher value than the starting value, indicating that after 24 hours waiting time preshearing does not reproduce the initial state. The 10 s and 10 min gel strength measurements are nearly unaffected by the preshearing. Comparison of Fig. 1 and 2, and Fig. 3 and 4, shows that preshearing gives more reproducible results than not preshearing, at least within a time frame of 6-8 hours after sampling.

Primary aim of the evaluation of the Fann 35 data is based on the high shear values (600 and 300 rpm). These two data points are in the field expressed by the Plastic Viscosity (PV) through the Bingham Plastic model, assuming a near constant

Figure 2. Fann 35 dial readings after indicated waiting time and 10 min preshearing. T= 28 ^{o}C. The lighter the colors, the longer the waiting times.

Figure 3. Fann 35 dial readings directly from resting after indicated waiting time. T= 50 ^{o}C. The lighter the colors, the longer the waiting times.
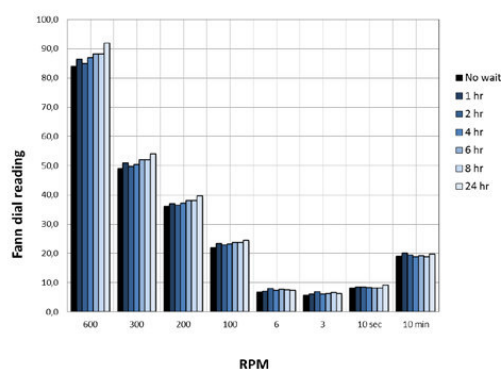

Figure 1. Fann 35 dial readings directly from resting after indicated waiting time. T= 28 ^{o}C. The lighter the colors, the longer the waiting times.
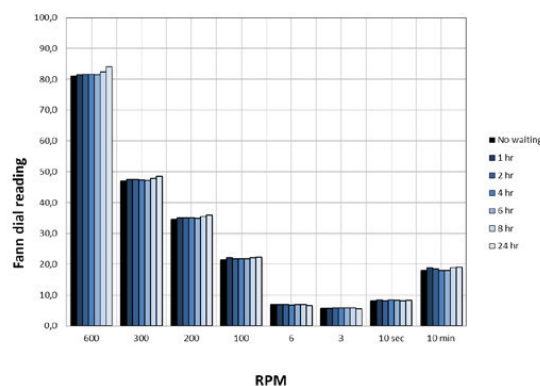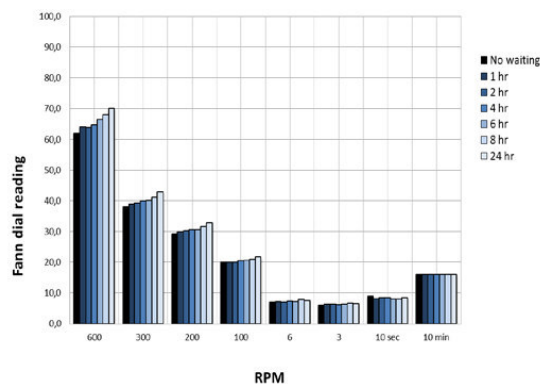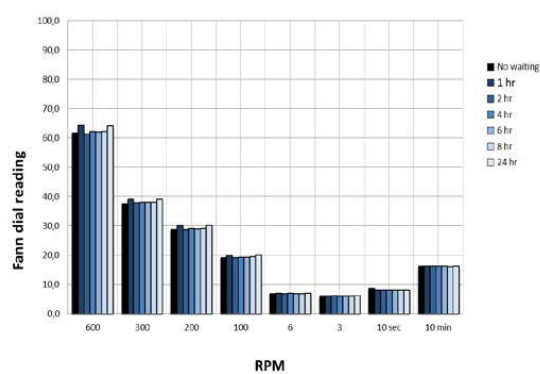

Figure 4. Fann 35 dial readings after indicated waiting time and 10 min preshearing. T= 50 ^{o}C. The lighter the colors, the longer the waiting times.
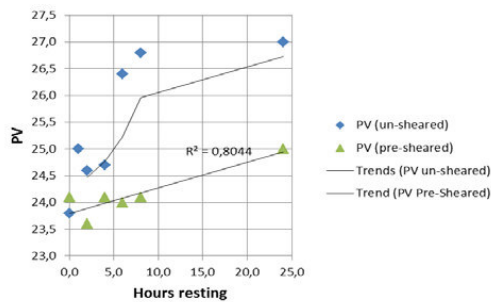
Figure 5. Plastic viscosity (mPas) plotted against resting time (h) for the non-presheared (diamonds) and the presheared (triangles) data, for 50 ⁰C. A linear fit is made to both data sets.

viscosity at high shear rates. The PV of a drilling fluid is described physically as the forces/friction between the interactions of non-continuous particles in the invert fluid, solids & brine droplets, creating a weak structure. Plotting the PV of the fluids towards the waiting time (see Fig. 5) shows for the 50 °C non-sheared measurements an increasing trend (3 whole digits) the first 8 hours and then a flattening trend up to 24 hours. For the 28 °C measurements, this flattening trend after some hours is not that evident (figure not shown). Preshearing removes this effect seen at 50 °C, but for both temperatures the presheared fluids show a linear increase in PV over 1 digit approximately (the 1 hour value at 50 ⁰C preshared disclaimed as an outlier). As the PV is believed to describe the friction of the particles at high shear, content, shape and, in particular, size will have an impact. It is not believed that the weighting material or other solids in this short period of time is aggregating to form larger sized particles. However, the internal phase consists of droplets that are highly attractive to each other, and kept dispersed in the continuous phase by the emulsifier in the system. If the emulsion is weakened over this period due to the lack of shear, the size of the internal phase droplets will increase due to aggregation of the water phase. This can

lead to a higher friction between the droplets and hence a higher PV.

Figures 6 and 7 show flow curves measured in the Anton Paar rheometer, at 28 and 50 ⁰C, respectively. The top bunch of lines in the figures represent flow curves with no preshearing. In Fig. 6, the structure build-up with no preshear at 28 ⁰C is clearly visible with increasing waiting times. The lower bunch of lines, representing measurements with preshear fall almost on top of each other, i.e. the same trend is apparent here as for the Fann measurements. The measurement for 8 hour rest and preshear clearly deviates from the rest and is considered an outlier.

Figure 7 shows the same measurements done at 50 ⁰C. At this temperature the structure regeneration with no preshear is much less pronounced, and the difference between the presheared and not presheared measurements is small. In other words, at 50 ⁰C preshearing has no significant influence on the flow curves. Figure 8 shows the storage (G') and loss modulus (G") of a strain sweep performed at a frequency of 10 Hz at 28 ⁰C, with no preshear. The measurement made immediately after mixing deviates from the others. Comparison with Fig. 9, showing the same measurement with 10 min preshearing at 1000 s^{-1}, reveals that preshearing gives more reproducible results, even after 24 hours waiting time. Preshearing has no effect on the G'/G" cross-over point (flow point) but the end of the linear viscoelastic range (LVER) is moved to higher strain values, i.e. the fluid tolerates a higher strain after preshearing before the structure starts to break down than it does with no preshear. For 50 ⁰C (Figs. 10 and 11), the picture is slightly different. Preshearing produces results much more similar than does no pre-shearing, but G'/G" cross-over point is moved significantly to higher strain values.
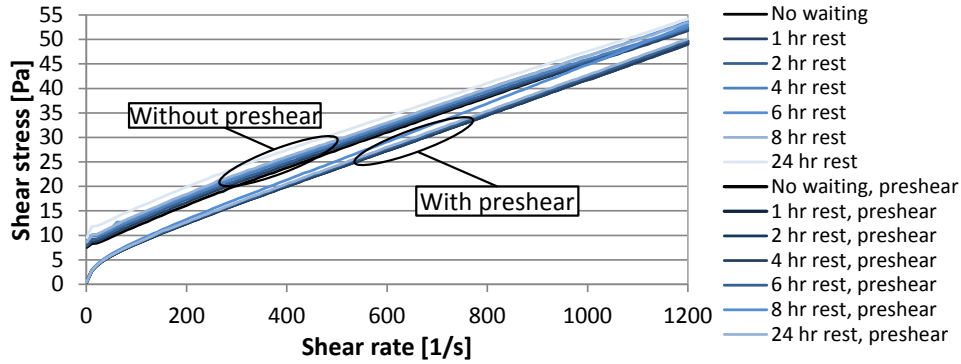
Figure 6. Flow curves of the OBDF for the non-presheared and the presheared samples at 28 ⁰C. The lighter colors represent longer waiting times.
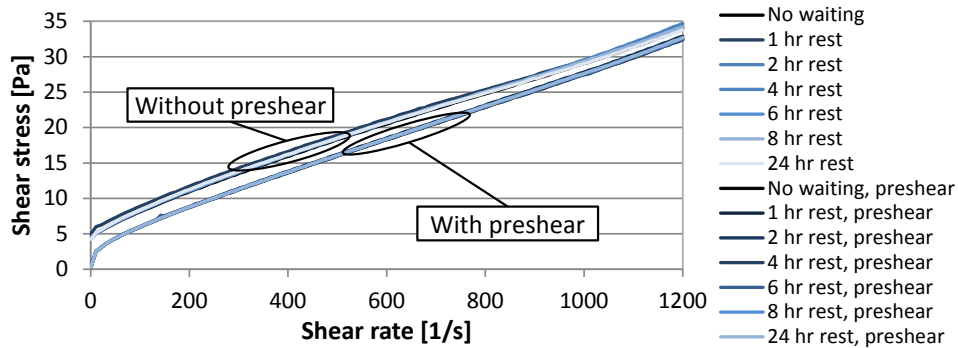
Figure 7. Flow curves of the OBDF for the non-presheared and the presheared samples at 50 ⁰C. The lighter colors represent longer waiting times.
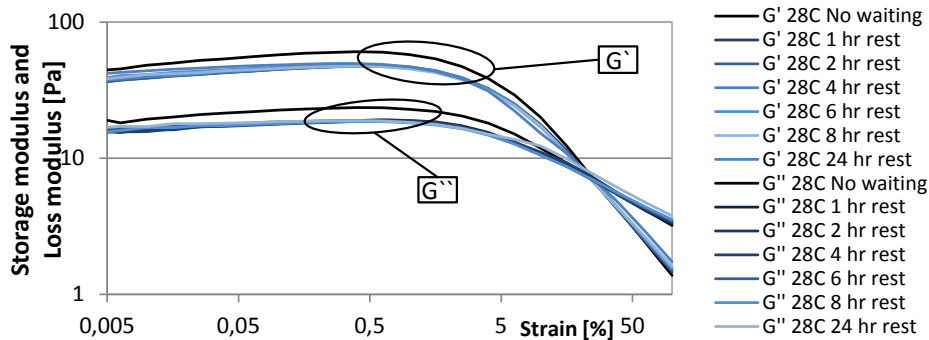
Figure 8. Amplitude sweeps of the OBDF for the non-presheared sample, at 28 ⁰C. Storage modulus (G') and loss modulus (G") are marked in the figure. The lighter colors represent longer waiting times.

End of LVER is also moved to higher strain values with preshear than with no preshear.

In other words, amplitude sweeps at 50 ⁰C are more sensitive to preshear than at 28 ⁰C.
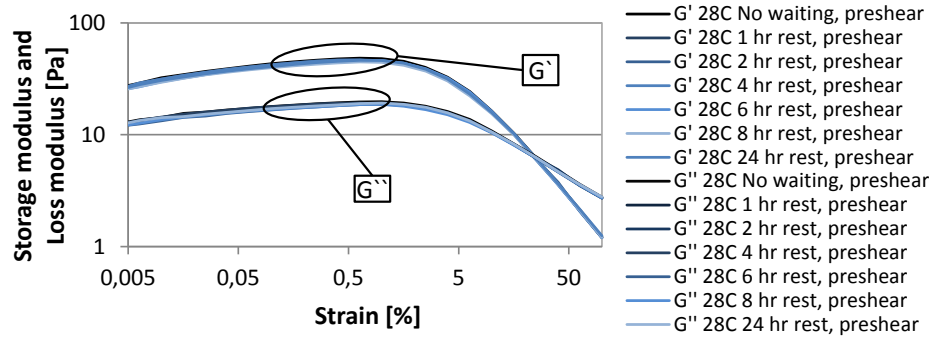
Figure 9. Amplitude sweeps of the OBDF for the presheared sample, at 28 ⁰C. Storage modulus (G')
and loss modulus (G") are marked in the figure. The lighter colors represent longer waiting times.
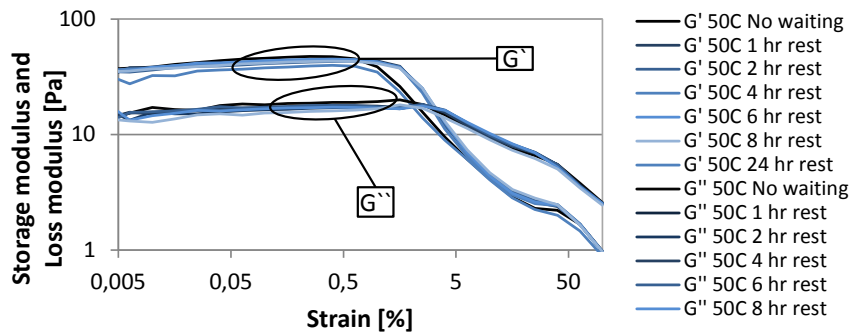
Figure 10. Amplitude sweeps of the OBDF for the non-presheared sample, at 50 ⁰C. Storage modulus
(G') and loss modulus (G") are marked in the figure. The lighter colors represent longer waiting times.
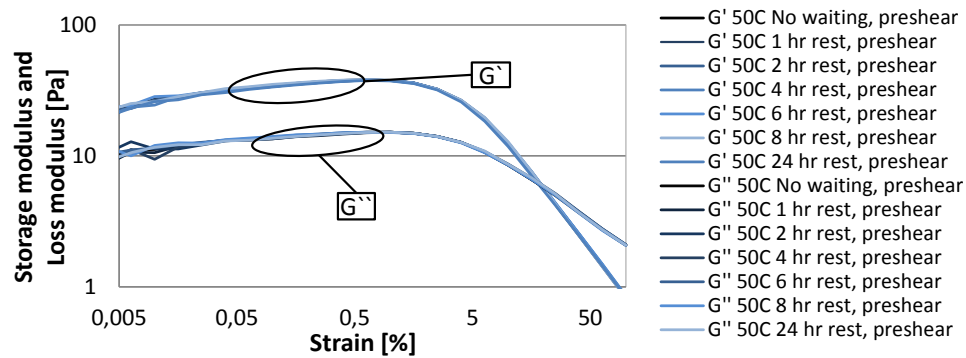
Figure 11. Amplitude sweeps of the OBDF for the presheared sample, at 50 ⁰C. Storage modulus (G')
and loss modulus (G") are marked in the figure. The lighter colors represent longer waiting times.

DISCUSSION
In this study, the samples have been
exposed to 10 min preshearing at 1000 s^{-1}.

The effect of preshearing for a shorter or
longer time or at a different shear stress has
not been tested. It is possible that shearing

for a longer time would give even more reproducible results[10]. At the same time, waiting before testing is a non-productive time, which would increase inefficiency and costs, especially in the field. Also, given that 10 min preshear is given in ISO/API standards, we suppose that 10 min preshear is a good compromise.

Results from this study are generally in accordance with expectations, although to our knowledge there are no publications so far where the effects have been systematically quantified. The results from this study are proposed as practical guidelines to measurements of viscosity and other rheological properties. The effect of waiting time and/or pre-shear should be tested for each individual fluid, but it is expected that these results will be valid for most oil-based drilling fluids.

CONCLUSIONS

For both 28 and 50 °C, one gets more reproducible results with preshearing, except for the 24 h measurements. Even with preshearing, sample readings change after 8 hours and/or 24 hours. Viscoelastic properties are even more sensitive to preshearing at 50 °C than at 28 °C, while this is not the case for purely viscous properties.

For comparative data, preshearing is recommended to achieve more reproducible results. For in-depth characterization of rheological properties, preshearing is not recommended as viscoelastic properties are significantly affected by preshear, especially for higher temperatures.

Most findings are according to expectations, but the effects are now quantified for both simple viscosity measurements (direct-reading viscometer) and for measurements of visco-elastic properties (rheometer). The findings may serve as a methodic reference work and a practical guide for rheological characterization of oil-based drilling fluids.

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Paper 2

Paper 3

Paper 4

Viscoelastic properties of drilling fluids and their influence on cuttings transport

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ARTICLE INFO

ABSTRACT

Cleaning the wellbore from drilled material is important to continue drilling efficiently and to prevent high torque and drag, as well as reducing down time due to reaming operations. Drilling fluids used for this purpose are complex fluid systems, generally with water or oil as a base substance. Water-based and oil-based drilling fluids are performing differently in terms of hole cleaning, even when their density and viscosity are fairly similar. Comparative studies performed by several research groups have resulted in diverse outcomes, showing superior behavior of either water- or oil-based drilling fluids, or no significant differences between the fluids at all.

In the present study, a water-based and an oil-based drilling fluid have been investigated regarding their visco-elastic properties, using the Anton Paar rheometers MCR 102 and MCR 302. Amplitude sweep tests, 3-intervall-thixotropy tests, temperature sweep tests, and low-shear rate flow curves with controlled shear stress and shear rate were performed and analyzed. Cuttings transport experiments in a flow loop with a 10 m long test section and a free-whirling inner-rotating drill string were conducted with the same fluids to study the hole-cleaning efficiency of different drilling fluids. The results from both experimental parts are presented. The rheometer results are used to interpret the cuttings transport behavior in the flow-loop experiments. The water-based drilling fluid was a KCl brine based fluid, and the oil-based fluid a water-in-oil emulsion. Both fluids are actual field fluids, used during drilling operations on the Norwegian Continental Shelf and have similar viscosities and densities.

The oil-based drilling fluid showed better hole-cleaning abilities during the flow-loop experiments, leaving a lower sand bed in the test section. This fluid displayed viscoelastic properties, such as a yield stress and a linear viscoelastic range. The water-based drilling fluid showed no yield stress and a 50–100% higher elasticity than the oil-based drilling fluid.

1. Introduction

Cuttings transport is an important part of every drilling operation. The removal of drilled material is necessary to avoid cutting accumulation in the borehole and proceed drilling. Optimal fluid properties will increase efficiency and effectiveness of the drilling operation (Aldea et al., 2005; Walker and Li, 2000; Becker et al., 1991). Oil-based (OBM) and water-based drilling fluids (WBM) are found to behave differently in terms of hole cleaning, even when density and viscosity are similar. The reasons for these differences are not fully understood. Ambiguous results on the superior performance of either OBM or WBM are reported in the literature. Several research groups have investigated causes, conducted experiments, evaluated field data, or compared fluid systems (Apaleke

et al., 2012; Bland et al., 2002; Li and Luft, 2014). The outcomes are diverse, spanning from results where oil-based drilling fluids perform better (Saasen and Løklingholm, 2002; Saasen et al., 1998), to results where water-based drilling fluids perform better (Hareland et al., 1993), to results where neither of the two types protrude (Hemphill and Larsen, 1996).

The rheology of drilling fluids not only influences the flow behavior of the fluids, but also the interaction with the cuttings and the cuttings bed, or in regard to the flow-loop experiments, the sand bed. Gelling effects of drilling fluids are important for their performance in the well bore (Herzhaft et al., 2006). Yield stress (Barnes, 1999; Møller et al., 2009) and thixotropy (Jachnik, 2005; Barnes, 1997) determine the suspension of cuttings when the pumping process is interrupted and the

drilling fluid is in static condition, for example during a tripping operation. Without the structure-building character of the fluids the suspended cuttings would start settling down towards the bottom of the wellbore, reducing hole-cleaning efficiency and leading to an accumulation of cuttings in narrow well sections risking higher drag and stuck pipe. The thixotropic characteristics describe the structure rebuild over time and are crucial for the suspension or settling of cuttings in static conditions. Yield stress and thixotropy are considered two different fluid characteristics, and are often present in the same fluids. The coupling between yield stress, viscoelastic properties and hole cleaning in the literature is presently rare.

To investigate viscoelastic properties of drilling fluids, or other materials, rheometers provide accurate measurement options. Oscillatory tests, like amplitude sweeps were performed to measure the short and long term structural behavior. Strain values are chosen small enough to measure the materials response, without destroying the structure in the material. Rotational tests were performed with either controlled shear rate (CSR) or controlled shear stress (CSS). Flow curves gained from CSS tests are used to find the yield stress of the material. The yield stress is the shear stress where the elastic deformation ends and the irreversible deformation starts. The value is dependent on the measuring method and/or the regression method and different approaches are widely discussed in the literature (Maxey et al., 2008; Møller et al., 2006; Stokes and Telford, 2004).

The experimental work presented here compares a water-based and an oil-based drilling fluid with similar density and viscosity. The aim of this study is to investigate the cuttings-transport behavior during flow-loop experiments with the help of rheological characterization of viscoelastic properties. Cuttings-transport experiments are performed in a flow loop with a free-whirling inner-rotating drill string. Results from these experiments are presented in Ytrehus et al. (2015). In the current investigation, amplitude-sweep tests, 3-intervall-thixotropy tests, temperature sweep tests, and low shear-rate flow curves with controlled shear stress and shear rate are performed and evaluated by use of state-of-the-art rheometers. The use of rheometers has gained importance due to the more advanced measuring possibilities compared to the Fann 35 viscometer measurements commonly used in the drilling industry (Schulz et al., 2013; Tehrani, 2007; Maxey, 2007, 2010; Clark, 1995). A focus of this study is a better understanding of the flow-loop results in light of the findings from the rheometer investigation.

2. Materials

The investigated KCl fluid is a water-based drilling fluid, where potassium chloride and glycol work as shale inhibitors and increase the density. Other additives are xanthan gum, to adjust the viscosity, polyanionic cellulose and starch for fluid-loss control, soda ash as a Ca^{2+} buffer, and barite as a solid weight material. The OBM is a water-in-oil emulsion with barite as a weight material. Organophilic clay, emulsifier, and a fluid-loss additive are other components dissolved or dispersed in the oil phase. Lime ($Ca(OH)_2$) is added to the water phase to assist the emulsifier, and $CaCl_2$ balances the activity of the formation water. The oil-water ratio accounted to 80/20 before the addition of the additives and the density was 1.26 g/cm^3 at 28 °C, which is slightly higher than for the KCl. Both fluids were delivered by MI-Swaco and are actual field fluids, used during drilling operations and reconditioned prior to the delivery to the research facilities. The components of both fluids are listed in Table 1 together with the density, oil/water ratio and Fann 35 viscometer values, measured in accordance with API (2010).

3. Experimental

3.1. Rheological experiments

To investigate the rheological properties of the introduced fluids, Anton Paar rheometers MCR 102 and MCR 302 were used in addition to

Table 1

Composition and comparison of OBM and KCl (Fann 35 data collected from Werner et al., 2016).

			OBM		WBM	
Components			Base oil EDC 95-11		Fresh water	
			Barite		KCl	
			Organophilic clay (Bentonite)		Glycol	
			Salt (CaCl₂)		Xanthan gum	
			Lime (Ca(OH)₂)		Polyanionic cellulose	
			Emulsifier		Starch	
			Fluid loss agent		Soda ash	
					Barite	
Oil-water ratio			Initially 80/20		Not applicable	
Density [g/cm³]			1,26		1,19	
Temperature [°C]			28	50	28	50
Fann 35	Shear rate [s⁻¹]		Dial readings			
	R₆₀₀	1,022	59	34	43	36
	R₃₀₀	511	34	19	32	27
	R₂₀₀	340,6	25	14	27	23
	R₁₀₀	170,3	15.5	9	20	17
	R₆	10,2	5.5	3	6.5	5
	R₃	5,1	5	2	5	4
Gel strength	10 s		–	3	5	4
	10 min		–	5	6	5

the Fann 35 measurements shown in Table 1. These rheometers are characterized by high precision and accuracy, and can be either shear or stress controlled. The conducted experiments include temperature sweep tests to study the temperature dependency of the fluids, amplitude sweep tests to estimate the storage and loss moduli and the linear viscoelastic range (LVER), which is necessary for the 3-interval-thixotropy tests. Controlled shear-stress sweeps are performed to estimate yield stress, and flow curves to find the relation of shear stress to shear rate. The flow curves of the OBM and the KCl based drilling fluids, as well as the controlled shear-stress curve and the temperature sweep of the OBM (Werner et al., 2016; Oltedal et al., 2015) are shown for comparison.

To assure repeatable results each test day, a procedure for pre-treating (Assembayev et al., 2015) the fluids before the experiments was established. The pre-treatment included shearing the fluids in a blender for 10 min at 6,000 rpm and afterwards letting them rest for 1 h. All experiments were conducted at 28 °C and at 50 °C, unless stated otherwise, and the temperature was controlled by a Peltier element. The temperature selection was based on two circumstances. 28 °C is the operational temperature of the flow loop and 50 °C is the proposed temperature for viscosity measurements following the International-Standard (2011), enabling a comparison to Fann 35 measurements. The CC27 (cylinder/cylinder) measuring geometry was chosen to minimize evaporation of sample at elevated temperatures over longer time periods. Thixotropy tests were also performed at 10 °C and 28 °C. A strain range of 0.001%–100% was used for the amplitude-sweep tests at a frequency of 10 1/s. The proposed strain value within the LVER from the software Rheoplus was then used for the 3-interval-thixotropy tests (3ITT). The 3ITT comprises three intervals. The first is a rest interval (low shear), followed by a high load interval, and finally a recovery interval (low shear). The first rest interval was 200 s of oscillation at a frequency of 10 1/s, conducted at 0.1% strain as the proposed software value within the LVER. During the high load interval a shear rate of 10 1/s was applied for 1 s. The recovery interval was set to observe the time needed until no structure rebuilt could be observed anymore. The oscillation frequency and strain were similar to the first interval.

Two types of low shear-rate flow-curve experiments were conducted. The first type of flow curves had a shear-rate range of 0.01 1/s to 100 1/s and the shear rate was ramped up logarithmically with a constant

measuring-point duration of 2 s. The second type was performed with a shear-rate range from 0.001 1/s to 100 1/s and was ramped up logarithmically with a logarithmic decrease in measuring-point duration from 30 s to 2 s. The latter procedure aims to reduce transient effects of the sample in the low-shear range for each measuring point due to the prolonged duration.

Experiments with controlled shear stress were done to estimate the yield stress of the fluids. During these tests the shear stress is increased logarithmically from 0.1 Pa to 100 Pa and the strain response is measured. The experiment stops when a flow regime is well developed and the yield stress is determined as the point where the stress-strain curve deflects from linearity.

The temperature-sweep tests were performed at a constant shear rate of 100 1/s in a temperature range of 5 °C–50 °C with a temperature increase of 1 K/min.

3.2. Flow loop experiments

The flow-loop experiments were conducted at an experimental rig with a 10 m long test section and a free-whirling inner-rotating drill string. The inner diameter of the test section is 100 mm and the drill string outer diameter is 50 mm. The flow velocities vary from 0.5 m/s to 1.2 m/s. A separation unit separates the fluid from the transported sand, enabling reuse of the fluid. The set-up (Fig. 1) is used to study cuttings transport and wellbore hydraulics of different drilling fluids to compare their hole-cleaning performance.

During the start-up of flow-loop experiments, a cuttings bed (sand bed) is created inside the test section. The sand is a quartz sand with particle sizes in the range of 0.9–1.6 mm, delivered by Dansand A/S. Drilling fluid is flowing with a velocity and sand is injected. The sand accumulates at the bottom of the annulus until a steady-state bed is reached. During the experiments the amount of sand leaving the test section, and the pressure gradients at different distances are measured. Experiments are conducted with and without drill-string rotation, at horizontal or inclined position. The operational temperature was 28 °C.

4. Results and discussion

4.1. Rheological experiments

Fig. 2a and b show a comparison of the storage and loss moduli from amplitude sweep experiments for the KCl and the OBM at 28 °C and 50 °C, respectively. The storage modulus represents the deformation energy which is stored in the sample during a shear process, and the loss modulus represents the deformation energy which is used during a shear process and lost for the sample (Mezger, 2014). The relationship between the loss modulus (G″) and the storage modulus (G′) is a measure of the stiffness of a material and the internal friction. Another related parameter is the linear viscoelastic range (LVER), which represents the strain range in which the G′ and G″ curves display constant plateau values, before the inner structure of the sample is broken. At 50 °C the storage modulus of the OBM develops a peak after the flow point, displaying an extra network structure (Mezger, 2014). The flow point represents the cross-over point of the storage and loss moduli, where G′ = G″. The storage modulus of the KCl fluid is lower than the corresponding loss modulus over the whole strain range and hence, no crossing point was found for neither of the temperatures. This indicates that the viscous behavior dominates the elastic. However, a clear flow point as well as LVER may be found where the G′ curve deflects from linearity. This behavior is typical for viscoelastic fluids including polymer solutions, where the molecular chains may be entangled, giving the fluid some structure, even though there is no consistent network of forces throughout the liquid. Table 2 shows the storage and loss moduli at the end of the LVER, their ratio (G″/G′), which is also called the loss factor, tan δ, and the upper limit of the LVER itself. When tan δ < 1, the sample shows a more elastic behavior, and respectively a more viscous behavior when tan δ > 1. The OBM showed tan δ < 1 for both temperatures and the KCl tan δ > 1. This is also displayed in Fig. 3, showing tan δ plotted over the strain range. When the curve deflects from linearity the internal friction changes.

The G′ values of the OBMs have a constant value until a strain of around unity is reached. This reflects that the fluid is constructed as a pure dispersion and emulsion. As long as the strain is less than around unity, the particle and droplet positions are kept in a position trying to reach an energy minimum. When the strain becomes larger the particles and droplets start to leap frog; totally destroying the structure and hence decreasing the G′ values. Here, the elasticity works on short range distances. The strain values of the WBMs show about 50–100 times higher values before the G′ curve starts to decrease. This indicates that the elasticity is not destructed by simply rearranging the position of the particles in the fluid.

The domination of the loss moduli over the storage moduli, and the corresponding loss factor of tan δ > 1 in the amplitude sweep tests for the KCl fluid indicate a viscous character. This is in accordance with other results presented here. The OBM shows a dominant elastic behavior, indicating a microstructure in the fluid.

Fig. 4 presents flow curves of the OBM and the KCl in a shear-rate range of 1–1,200 1/s for both tested temperatures. The yield stress of the two OBM curves can be seen as the crossing point with the y-axis. Both fluids seem to be most shear thinning in the lower shear rate range and later increasing much more linearly. This accounts for both temperatures. The OBM is clearly more affected by rise in temperature than the WBM.
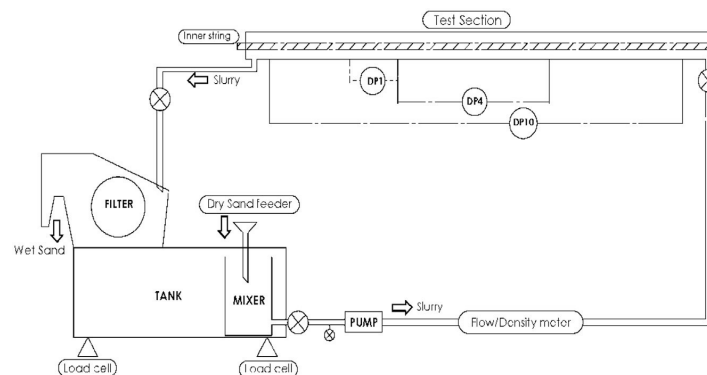

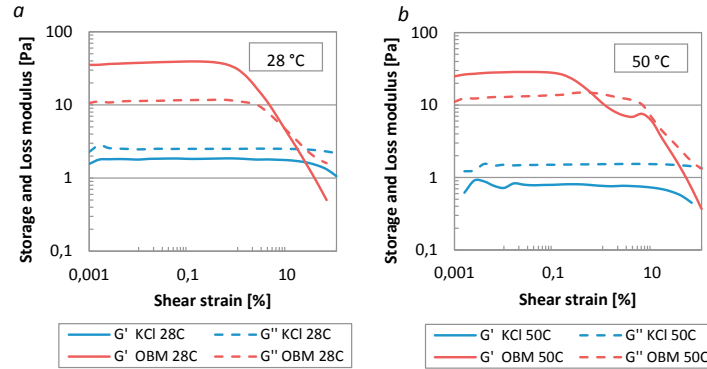**Fig. 1.** Schematic figure of the flow loop set up.

Fig. 2. (a) Amplitude sweep showing storage and loss moduli of the KCl and OBM fluids at 28 °C, (b) Amplitude sweep showing storage and loss moduli of the KCl and OBM fluids at 50 °C.

Table 2
Storage and loss moduli, loss factor, and LVER of OBM and KCl for 28 °C and 50 °C.

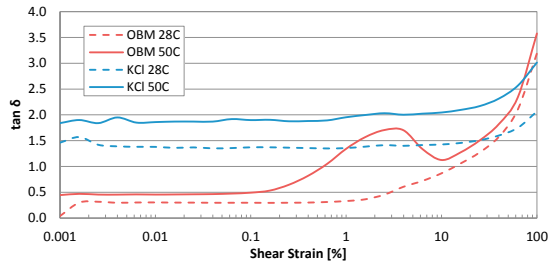| Fluid	Temperature [°C]	G' [Pa]	G'' [Pa]	tan δ	LVER (%/Pa)
OBM	28	37.6	11.3	0.3	0.02/37.6
	50	27.8	13.7	0.5	0.1/27.8
KCl	28	1.8	2.5	1.4	6.3/1.8
	50	0.8	1.5	1.9	6.3/0.8

Fig. 3. Ratio of the loss to the storage moduli plotted over the shear strain range.
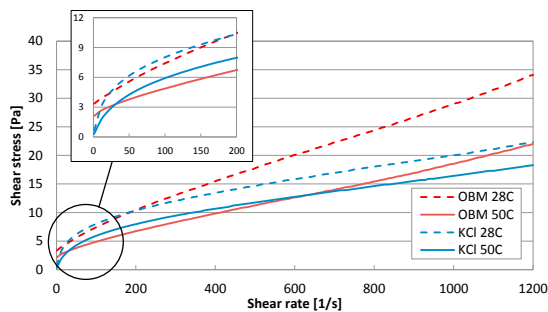

Fig. 4. Flow curves in a shear-rate range of 1–1,200 1/s for OBM and KCl at 28 °C and 50 °C (OBM data collected from Oltedal et al., 2015).

In Fig. 5 low shear-rate flow curves in a shear-rate range of 0.001–100 1/s are presented. During these experiments the measuring-point duration was logarithmically decreased throughout the experiment from 30 s to 2 s from first to last measuring point. For both temperatures, the OBM shows a peak and a plateau. For the 50 °C measurement, the peak is in the
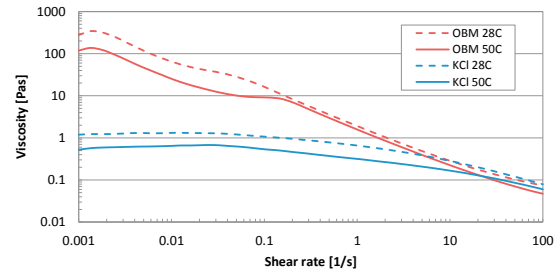

Fig. 5. Low shear-rate flow curve in a shear-rate range of 0.001–100 1/s with decreasing measuring-point duration of 30 s–2 s for OBM and KCl at 28 °C and 50 °C.

very low range at 0.001 1/s and the plateau at a shear rate of 0.1 1/s. For the 28 °C measurement the peak is at 0.001 1/s as well, but the plateau is moved to the left and is less predominant than for the 50 °C measurement. The KCl fluid does not show any clear peaks for neither of the two investigated temperatures, but both curves have an inflection point at 0.03 1/s. The inflection point is slightly higher for 50 °C than for the 28 °C measurement.

The low shear-rate flow curves of the OBM show a shear-thickening tendency at very low shear rates of about 0.001 s^{-1}. A similar peak appeared after redoing the experiment and starting from a shear rate of 0.01 s^{-1} (see Fig. 6). The fact that the peak shows up also at a different starting shear rate indicates that the increase in viscosity is not tied to a particular shear rate but is rather a transition effect of the fluid going from still stand to moving. It may be an effect of the tendency of water droplets to arrange themselves in certain patterns depending on the
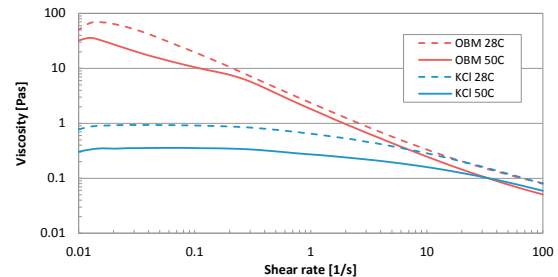

Fig. 6. Low shear-rate flow curve in a shear-rate range of 0.01–100 1/s with constant measuring-point duration of 2 s for OBM and KCl at 28 °C and 50 °C.

conditions in the dispersion. In static conditions Brownian forces may be responsible for positioning the water droplets of the emulsion in an arrangement with the furthest possible distance between each droplet, resulting in a crystal like structure and high viscosity. A transition zone between the shear rates of $0.05 \, s^{-1}$ and $0.2 \, s^{-1}$ is dividing the curves into two plateau like areas. Similar behavior was seen by Herzhaft et al. (2002), where a transition between Newtonian behavior at low shear rates and non-Newtonian behavior at higher shear rates was found. Another explanation could be that for very low shear rates the Brownian motion positions the water droplets in a more regular structure in the mean, which then appears as crystalline structures (Ackerson, 1990). Saasen (2002) extended this explanation during a study on barite sag in oil-based drilling fluids with the following conclusion. The crystalline structure remains intact, as long as the Brownian motion is dominating. With increased shear rate, the Brownian motion is not able to reconstruct the crystalline structure of the water droplets any longer. Individual droplets then have to let other droplets redirect themselves, resulting in a chaotic motion and a significant increase in viscosity leading to the peak seen at very low shear rates. A deeper investigation is needed to fully understand the behavior. Changing the measurement direction to tests conducted from high to low shear rates could be a possibility.

Fig. 6 shows low-shear flow curves at a shear-rate range from 0.01 to 100 1/s, using a constant measuring-point duration. At 50 °C the OBM develops a small plateau at a shear rate of 0.013 1/s. At a shear rate of 0.2 1/s, the curve displays a sudden change in slope. After the slope change the curve continues almost linearly. At 28 °C the plateau is visible at a shear rate of 0.02 1/s and the analysis shows a slight slope change at about 0.03 1/s. The KCl does not show any clear plateaus or peaks and the viscosity values are lower for low shear stresses compared to the OBM. This accounts for both temperatures. From about 10 1/s the KCl starts to approach the OBM curves.

The overall effect of changing the measuring-point duration from logarithmic decreasing to constant, is to smooth out the curves. The reason for this is probably that at constant measuring-point duration, the duration is too short to gather significant amount of data especially for the low shear rate measurements at low torque. Hence, some features of the curves are lost.

Fig. 7 shows controlled shear-stress sweeps in a range of 0.1–10 Pa. The two curves for the OBM show a clear yield stress at the deflection from linearity, marked by diamonds. The slight deflection before 1 Pa is probably due to a transition state and can be disregarded. The yield stress for the 28 °C measurement is higher than for the 50 °C measurement. The KCl fluid develops a linear plateau and does not deflect from that in the higher shear-stress area. This is consistent with findings from the amplitude sweeps. The controlled shear-stress sweeps for the KCl fluid show no deflection from linearity, therefore no yield stress can be determined. This is in conformity with the results from the amplitude-sweep tests, where the crossing point (flow point) of the storage and loss moduli can be interpreted as a yield stress, and the flow curves. During the current measurements no crossing point was found due to the dominating loss modulus, hence no yield stress could be determined either. The decrease of viscosity and elasticity with increased temperature was also observed by Bui et al. (2012).

Temperature-sweep curves are presented in Fig. 8. The sweep is performed from low to high temperatures. The viscosity of the KCl fluid decreases almost linearly throughout the whole tested temperature range. The viscosity of the OBM decreases quite non-linear with a relatively steep decrease in the beginning, and starts to flatten out for higher temperatures. A reason for this behavior could be the base oil in the OBM. It consists of long chained hydrocarbons (C16 or higher), which may start crystalizing at lower temperatures, hence increasing the viscosity at lower temperatures. At higher temperatures the expansion character of the oil phase increases its volume and influences other parameters.

Fig. 9 shows the storage and loss moduli for the 3-interval-thixotropy tests of the KCl fluid at 10 °C, and 28 °C. Those temperatures are chosen because they represent each side of the crossing-point temperature
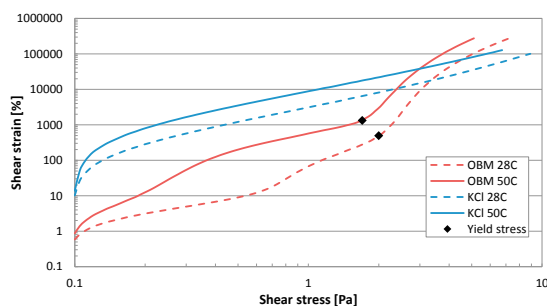

Fig. 7. Controlled shear-stress sweeps from 0.1 to 100 Pa (log) for OBM and KCl at 28 °C and 50 °C (OBM data collected from Oltedal et al., 2015).
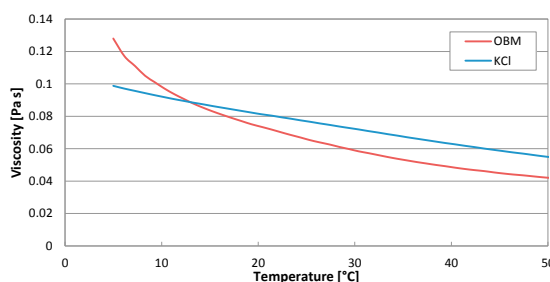

Fig. 8. Temperature-sweep tests at a shear rate of 100 1/s in a temperature range of 5–50 °C for OBM and KCl (OBM data collected from Oltedal et al., 2015).
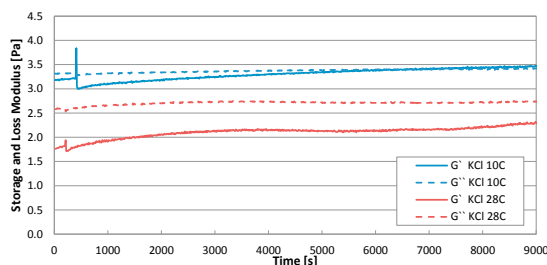

Fig. 9. 3-Interval-Thixotropy test for the KCl at 10 °C and 28 °C.

shown in Fig. 8. The loss moduli are dominating the storage moduli during the rest interval and the beginning of the recovery interval, similar to the amplitude sweep tests. For the 10 °C and the 28 °C tests the storage moduli approach the loss moduli and in the 10 °C test the storage modulus is crossing the loss modulus after about 6,500 s. This means that for the 10 °C test, after about 6,500 s the structure of the sample is rebuild to the extent that the elastic behavior is now dominating.

A clear crossing point was not possible to determine exactly, because in the range of 6,000 s–8,000 s the two curves are crossing each other several times. At a temperature of 28 °C the fluid structure seems to be more weakened to the point that the structure rebuilding is more moderate, at least within the timeframe of our experiment (9,000 s = 150 min). This finding may be of significance to drilling operations with regards to the fluids capability to rebuild structure and keep cuttings suspended during down times.

4.2. Flow loop experiments

In Fig. 10 the sand holdup versus the superficial liquid velocity for the KCl and the OBM are presented (Experimental data by Sayindla et al. (Submitted)). Results are shown for experiments in horizontal position without drill-string rotation. The sand holdup is the height of sand bed left in the (eccentric) annulus in percent of the total diameter, after steady state condition at a specific fluid velocity was reached. The sand holdup is determined by calculating the difference of the input-sand mass and the output-sand mass of the test section. The input sand rate is 43 g/s. The influence of the flow velocity is visible in Fig. 10, i.e. higher flow velocities remove more of the sand bed than lower flow velocities. At a fluid velocity of 1.2 m/s without drill-string rotation, the OBM leaves 5.8% and the KCl 10.6% of the sand-bed hight in the test section. The effect of drill-string rotation was also tested. In two sets of experiments with the two fluids and a drill-string rotation of 150 rpm, a high impact on cuttings removal for both fluids was observed, but no significant difference in sand holdup between the two fluids could be seen. The drill-string rotation being dominating over the flow-related properties in regard to cuttings transport (Sayindla et al., 2016). The rotation destroys the particle interactions, reducing the resistance of the sand bed and helping the sand particles getting suspended into the fluid flow and transported out of the test section. Turbulences created by the rotation increase particle motion and counteract the settling of sand particles.

To set the flow-loop experiments into perspective to the rheometer and Fann 35 measurements, the relevant shear-rate range for experiments without drill-string rotation is estimated using equation (1), where D is the borehole diameter, d the drill-string diameter, v the fluid velocity, n the flow index, R the borheole radius, r the drill-string radius, and ω the drill-string rotation. The equation does not count for yield-stress fluids.

$$\dot{\gamma} = \left[\left(\frac{12v}{D-d} \frac{2n+1}{3n} \right)^2 + \left(\frac{\omega r}{R-r} \right)^2 \right]^{\frac{1}{2}} \qquad (1)$$

The highest shear rate for a fluid velocity of 1.2 m/s accounts to 295 1/s. In a Fann 35 viscometer, a shear rate of 295 1/s occurs at a rotational speed of 173 1/min.

The experiments without drill-string rotation, see Fig. 10, show a lower sand holdup for the OBM at all tested fluid velocities. The biggest difference between the OBM and the KCl fluid is at a velocity of 1.2 m/s, the highest velocity tested, pointing out the influence of flow velocity on hole cleaning. The OBM removes more sand out of the test section than the KCl fluid at all tested fluid velocities, and can for this experimental campaign be considered the better fluid for cuttings transport. The OBM
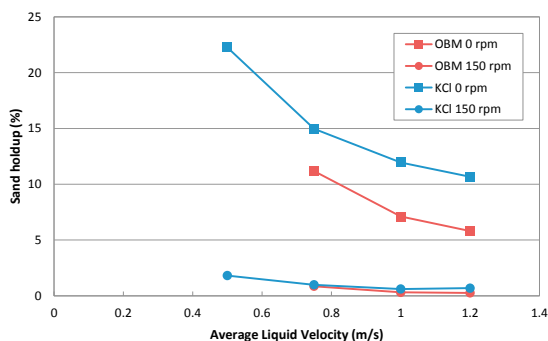
is showing a clear yield stress in the CSS tests and elastic dominated behavior in the amplitude-sweep tests, in contrast to the KCl, which showed a viscous dominated behavior over the elastic part. When comparing the flow-loop results, the OBM removed a higher percentage of sand bed out of the test section, compared to the KCl fluid. The superior behavior of OBM can possibly be explained by better suspension of the sand particles in the fluid, hindering or decelerating the settling due to the microstructure. Similar behavior was reported by Saasen (1998) and xanthan gum highlighted as an explanation. The gel-structure build up induced by the polymer can add more resistance to the sand bed and therefore cause less effective cuttings transport. Water-based drilling fluids are mainly clay suspensions, viscosified with polymers. The microstructure exhibited in a state of rest can be explained by interactions of the suspended particles, due to Brownian motion. Polymers, water and solid particles may create strong inter-particle forces, increasing the interactive forces of the sand bed and the sand particles, creating more resistance to cuttings transport. The polymers in the WBM tolerate more elastic deformation (strain). This can be seen in the amplitude sweeps (Fig. 2a and b). The strain values for KCl are higher than for OBM by a magnitude of about 100 before G' starts to decrease. During drill-string rotation, the elasticity in the fluid will assist the movement of the cuttings from the bed around the drill string and into the fluid flow. The 3-interval-thixotropy tests from Fig. 9 may suggest a similar behavior. For the 28 °C experiment the storage modulus is increasing over time and approaching the dominating loss modulus, giving the impression of a structure buildup. The test time was not long enough to see a cross-over point in this measurement. During the 10 °C experiment a cross-over point can be seen, meaning the KCl established elasticity-dominated behavior after a longer period in static condition.

5. Conclusion

An oil-based and a water-based drilling fluid have been compared regarding their rheological properties and their hole-cleaning capabilities. The characterization was done with an Anton Paar rheometer measuring flow curves, amplitude sweeps, temperature sweeps, and thixotropy. Hole-cleaning capabilities were investigated with flow loop experiments.

The oil–based drilling fluid used in this study showed a superior cuttings-transport ability to the water-based drilling fluid. The rheological characterization of the tested fluids provided insights into their viscoelastic behavior. The results suggest particle and emulsion based fluids exhibiting light internal structures at low shear rates and small yield stresses being the better option for hole cleaning, most likely due to a better suspension capability of the cuttings in the fluid during flow, and due to the absence of polymers consolidating the cutting beds that make these beds difficult to remove.

Using a rheometer for the rheological analysis of drilling fluids is a good way to enhance the understanding of the low shear behavior, structural properties, and time dependence.

Acknowledgments

Fig. 10. Sand holdup versus superficial liquid velocity for KCl and OBM with and without drill-string rotation (OBM data collected from Sayindla et al., (2016), KCl data from Sayindla et al. (Submitted)).

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Paper 5

Effect of Preconditioning and Ageing on Rheological Properties of Model Drilling Fluids

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ABSTRACT

Many drilling fluids are thixotropic, meaning that viscosity decreases with time when subjected to shear. Other fluids may show anti-thixotropic (rheopectic) behavior, i.e. viscosity increase with time when subjected to shear. In both cases it is important to have a consistent procedure for how to treat the fluids prior to rheological measurements. This is vital in order to be able to compare measurements of different samples, with different instruments and between different laboratories.

In the oil industry, the ISO 10416/ISO 10414-1/2 standards are used for determination of viscosity and gel strength of fluids by use of direct-indicating viscometers (Fann 35 viscometers). However, these standards do not specify in detail how the fluids should be pre-treated before measurements. Further, if results from Fann 35 viscometers are to be compared to measurements done on a rheometer, it is even more important to have a consistent pre-treatment of the fluids before the measurements.

A systematic study is performed in which the effects of pre-shearing and ageing history on the rheological measurements of model drilling fluids are investigated by using a Fann 35 viscometer and an Anton Paar MCR 302 rheometer.

The results demonstrate the importance of consistent preconditioning of fluids before measurements. A test-procedure standard is proposed, enabling higher measurement precision and comparability of rheological measurements.

INTRODUCTION

Thixotropy is defined as the decrease with time of viscosity under constant shear rate or shear stress, followed by a gradual recovery when the shear rate or shear stress is removed[1]. Thixotropy is thus a reversible property, and many oil-field drilling fluids exhibit thixotropic properties as they are designed to be shear-thinning. However, rheological properties of drilling fluids may also change irreversibly over time due to degradation of the fluids over time due to pressure, temperature, shear history etc, and due to contamination with other fluids, particles and chemicals.

It is therefore important to define consistent measurement methods, which can distinguish between viscosity changes caused by these different effects.

Thixotropy originates from the microstructure of these complex fluids, and the state of this microstructure is dependent on the history of the fluid (shear stress/rate, pressure, temperature, etc). In order to be able to make reproducible experiments it is therefore necessary to recover the original microstructural state of the fluid. Different procedures have been defined in order to achieve this. A typical procedure is to preshear the fluid at some high shear rate

immediately before the measurement, sometimes after a specified waiting time.

Aqueous solutions of laponite are good substances to research this problem with, as they show similar characteristics to water-based drilling fluids. They are ageing, that means their relaxation time increases with time, shear thinning, and show thixotropic behaviour[2].

In a previous paper Assembayev[3] et al. studied the effect of preshearing and waiting time on Fann 35 and Anton Paar measurement results for an oil-based drilling fluid. It was found that with preshear at 1020 s[-1] for 10 minutes the results were significantly less dependent on the prior history (waiting time) than without preshear for waiting times of about 8 hours or less.

In this paper we present and interpret experimental results from Fann 35 and Anton Paar measurements on different water-based model fluids.

The purpose of the paper is to investigate the impact of pre-treatment (preshear and waiting time) for different water-based model fluids, and how this is affected by salinity, temperature and addition of polymer.

FLUIDS

Five different fluid compositions have been tested. All fluids are aqueous solutions of Laponite RD. Laponite is a synthetic clay with disc-like particles. Its chemical formula is $Na^+_{0.7}[(Si_8Mg_{5.5}Li_{0.3})O_{20}(OH)_4]^-_{0.7}$. A negative surface charge is generated from a substitution of magnesium atoms by lithium atoms, to counterbalance the positive charge of the sodium ions[4]. In aqueous media, the sodium dissociates, leading to a negative charge on the surface[5]. Aqueous suspensions of laponite usually show a rich variety of physical behaviour.

The fluid-mixing procedure started with hydrating the laponite in deionized water for 24 h. Afterwards xanthan gum, NaCl, NaOH and biocide were added in varying concentrations, as shown in Table 1 together with the pH and conductivity values.

EXPERIMENTAL

Measurements were conducted using a Fann 35 viscometer and an Anton Paar MRC 302 rheometer, using a concentric cylinder (CC27) configuration. The rheometer is equipped with a peltier element to heat the sample to the desired temperature. The Anton Paar measurement procedure was as follows: The fluids were sheared for two minutes in a Waring blender at low speed and left to rest for one hour to assure an equal starting point for each fluid. For experiments with preshear the sample was placed in the measuring cup and sheared for 2 min at 1020 1/s (600 rpm in Fann 35 viscometer). Immediately after the preshear interval, the flow curve measurement was started with a decreasing shear rate from 1020 1/s to 1 1/s. Tests without preshear were conducted in the same way, but without a preshear interval. The procedure was repeated after a waiting time of 24 hours. The Fann 35 measurements follow the ISO 10414-1 standard[6]. Six measurements were taken at distinct rotational speeds of 600, 300, 200, 100, 6, and 3 rpm.

RESULTS AND DISCUSSION
Fann 35 measurements

Generally, the fluids show shear thinning trends, and experiments with preshear and without waiting time led to the lowest viscosities. The biggest difference in viscosity can be seen between the measurements without waiting time and a waiting time of 24 h, especially for fluids #1a and #2a. Small amounts of salt increase the viscosity, this applies both with and without xanthan gum present in the composition, see Figure 1 - Figure 4.

A high salt concentration of 12 g/l NaCl reduced the viscosity of sample #2c, compared to #2b (Figure 5).

Figure 1 - Figure 4 show that the preshearing procedure is not sufficient to obtain results independent of waiting time for fluids #1 and #2. However, preshearing is much more efficient when 0.6 g/l NaCl is added, as seen in Figure 2 and Figure 4. The

measurements with fluid #1a and #1b were also conducted at 50 °C and show the same qualitative trends as at 24 °C.

Interestingly, the experiments with fluid #2, which include xanthan gum, show similar trends as fluids #1. For fluids without salt and measurements without preshear the viscosity is much lower.

As expected, preshear does not have much effect on the result with 0 hr waiting time.
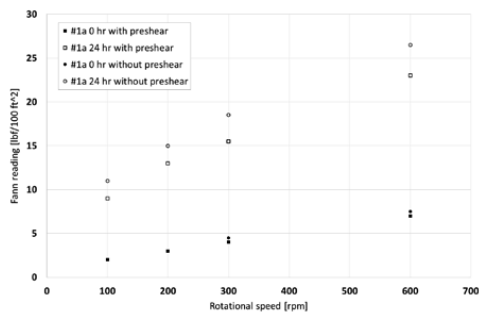

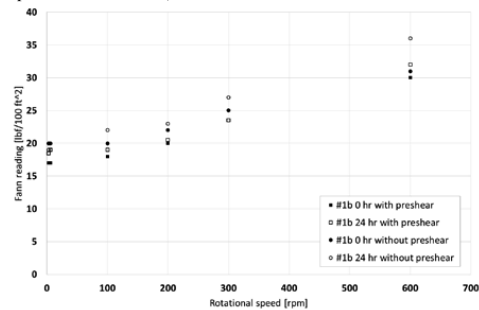*Figure 1. Fann 35 measurements for fluid #1a (water-laponite without salt) at 24 °C.*

Figure 2. Fann 35 measurements for fluid #1b (water-laponite with 0.6 g/l NaCl) at 24 °C.
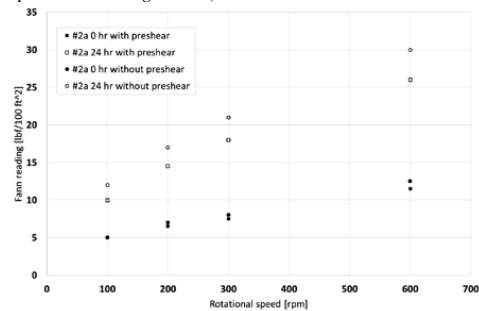
Figure 3. Fann 35 measurements for fluid #2a (water-laponit-xanthan gum without salt) at 24 °C.
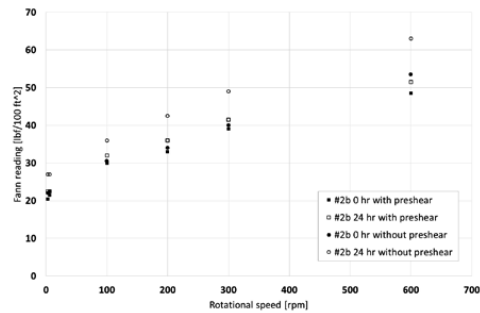

Figure 4. Fann 35 measurements for fluid #2b (water-laponit-xanthan gum with 0.6 g/l NaCl) at 24 °C.
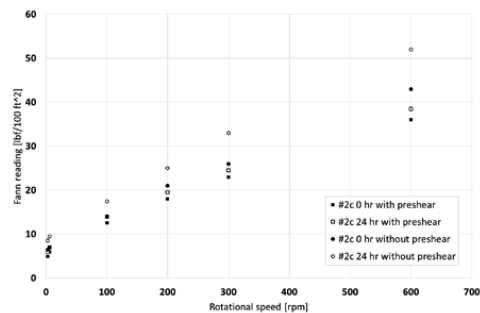

Figure 5. Fann 35 measurements for fluid #2c (water-laponit-xanthan gum with 12 g/l NaCl) at 24 °C.

Anton Paar measurements

The observations made from the Fann measurements are confirmed by the flow curves generated from Anton Paar measurements.

The flow curves measured with the Anton Paar rheometer show apparent shear thickening trends in the samples without preshear for shear rates >800 1/s (Figure 7). This trend is more pronounced for samples with a waiting time of 24 h and higher NaCl concentrations and especially predominant for fluid #2c, which contains xanthan gum and a high concentration of salt. This effect might be a result of the thixotropic behaviour, because the measurements start at high shear rate and are affected by a shear history.

In Figure 6 the curves without waiting time show a kink at a shear rate of about 450 1/s. The kink was observed regardless of measuring direction and at 50°C. We do not

have a consistent explanation, because we see it only in the Anton Paar measurements and not in the Fann 35 measurements. The Taylor number for this case was well below the critical Taylor number, see Equation 1, where ω is the rotational speed, b the radius of the bob, a the radius of the cup and ν the kinematic viscosity.

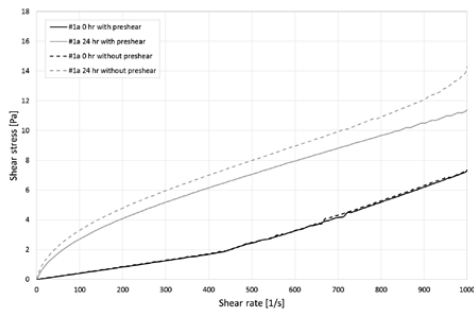$$Ta = \frac{\omega^2 b (a-b)^3}{\nu^2} \qquad (1)$$

Figure 6. Anton Paar flow curve measurements for fluid #1a (water-laponite without salt) at 24 °C.
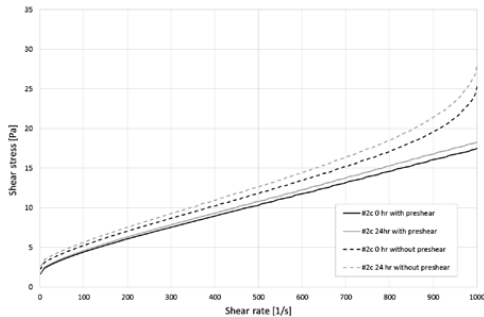

Figure 7. Anton Paar flow curves of fluid #2c (water-laponite with salt and xanthan gum) at 24 °C.

Figure 8 shows a comparison of Fann 35 and Anton Paar measurements of fluid #1a at 24 °C. The results show a difference for the measurements without waiting time. The shear stress of the Fann 35 measurement is much below the shear stress value of the Anton Paar measurement at high shear rate. It is possible that this can be a geometry effect, although we then would expect to see this in other measurements as well.
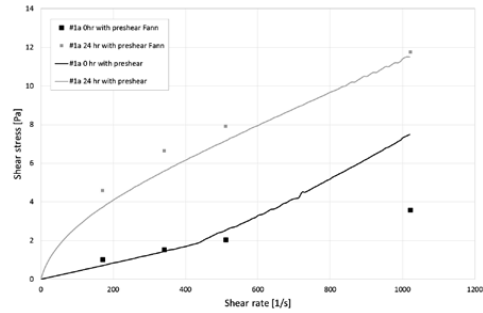

Figure 8. Comparison of Fann 35 and Anton Paar measurements for fluid #1a (water-laponite without salt) at 24 °C.

CONCLUSION

Preshearing a thixotropic sample before the measurement of flow curves is recommended to achieve more reproducible results. Preshearing is not recommended when measuring structural properties, as preshearing will reduce or destroy the microstructure in the fluid. The findings work as a methodical guideline for rheological characterization of water-based model fluids. The effects of waiting time and/or preshear are significant and will influence the quality of the results. Salt affects the influence of preshear, both with and without xanthan gum. Preshear is much more effective with a small quantity of NaCl than without NaCl. The effects of concentration of salt on viscosity and on the effect of preshear is strongly nonlinear, however we have only investigated two different salt concentrations. Xanthan gum alone does not have this effect. We see the same qualitative trends for 50 °C as for 24 °C. An anomaly was found in the Anton Paar measurements with a laponite fluid without salt. This was consistent but not seen in the Fann 35 measurements. Also the Taylor number is well below the critical Taylor number for this fluid.

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Table 1 Fluid composition

Component	1		2		
	a	b	a	b	c
Water	Deionized water				
Laponite RD [wt %]	1.5	1.5	1.5	1.5	1.5
Xanthan Gum [wt %]	0	0	0.1	0.1	0.1
NaCl [g/l]	0	0.6	0	0.6	12
NaOH [mmol/l]	0.1	0.1	0.1	0.1	0.1
Biocide	0	0	0.1	0.1	0.1
pH value	10.07	10.31	10.51	10.21	9.31
Conductivity [mS]	0.576	1.525	0.546	1.382	17.7